

# NASA Technical Memorandum 89607

## Microgravity Science and Applications Program Tasks

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# Microgravity Science and Applications Program Tasks

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## **I. INTRODUCTION**

## NASA TECHNICAL MEMORANDUM

### I. INTRODUCTION

The Microgravity Science and Applications (MSA) Program is directed toward research in the science and technology of processing materials under conditions of low gravity to provide a detailed examination of the constraints imposed by gravitational forces on Earth. The program is expected to lead, ultimately, to the development of new materials and processes in commercial applications adding to this nation's technological base. The research studies emphasize the selected materials and processes that will best elucidate the limitations due to gravity and demonstrate the enhanced sensitivity of control of processes that may be provided by the weightless environment of space. Primary effort will be devoted to a comprehensive study of the specific areas of research which revealed potential value in the initial investigations of the previous decades. Examples of previous process research include growth of crystals and directional solidification of metals in the quiescent conditions in which gravitational fluid flow is eliminated; containerless processing of reactive materials to eliminate reactions with the container and to provide geometrical control of the product; synthesis and separation of biological materials in weightlessness to reduce heat and mass transfer problems associated with sedimentation and buoyancy effects; identification of high vacuum characterization associated with an orbiting wake shield; and minimal knowledge of terrestrial processing methods.

Additional effort will be devoted to identifying the special requirements which drive the design of hardware to reduce the risk in future developments. Examples of current hardware studies are acoustic, electromagnetic, and electrostatic containerless processing modules and electrophoresis separation devices.

The current emphasis on fundamental processing science and technology in selected areas will continue as the Microgravity Science and Applications Program addresses problems of interest to the public and private commercial sectors which can be resolved by recourse to the space environment.

Emphasis will be placed on the expansion of currently funded activities for ground-based and space flight investigations to maximize the outputs from these opportunities. Initiatives requiring new hardware will be encouraged at a low level until funds can be made available. The expansion of current efforts is occurring as a result of focusing support for current space flight investigations on forming

facility experiment teams to provide advice and identify future involvement. Emphasis has been placed on experiments involving the Materials Experiment Assembly and Mid-deck experiments on the Space Shuttle.

The Microgravity Science and Applications Program Task Document covers the period of May 1985 - November 1986, (FY86 and part of FY87). The document includes research projects already completed as well as those now being funded by the Office of Space Sciences and Applications, Microgravity Science and Applications Division, NASA Headquarters.

## **II. TASKS**



**A. GROUND BASED EXPERIMENTS**

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**1. ELECTRONIC MATERIALS**

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## Processing of Complex Compound Semiconductors in Space

North Carolina State University

Dr. K. J. Bachmann

Dr. K. Y. Lay

NAG1-354

May 1, 1983 - April 31, 1986

In the past two one-half years of ground based research we have studied growth of bulk II-VI alloy and III-V single crystals from the melt by the modified Bridgman method and zone leveling. The materials selected for this study:  $Mn_xCd_{1-x}Te$ ,  $Zn_xCd_{1-x}Te$ ,  $CdSe_yTe_{1-y}$ ,  $Cd_xHg_{1-x}Te$  and  $InAs_ySb_{1-y}$ ; are of interest in the context of far infrared detectors and optical imaging devices for satellite based surveillance systems.  $Mn_xCd_{1-x}Te$  is of additional interest as a magnetic semiconductor and, out of the above group of materials, has been thoroughly characterized by us. XPS studies on cleaved and/or sputtered surfaces oxidized in a controlled UHV environment reveals that Mn segregates in the native oxide and that, as for pure CdTe, the charge state of  $Te^{2-}$  changes in the oxide to  $Te^{4+}$ . Photorefectance (PR) and photoconductivity (PC) measurements resulted in data on the band gap as a function of composition and temperature in the range of  $10K < T < 300K$ . The broadening of the PR signal reveals that alloy broadening is the dominant mechanism up to  $x < 0.2$ . At  $x \sim 0.3$  the broadening increases discontinuously which is related to faceted growth accompanied by microtwinning. The same behavior is observed in the low temperature photoluminescence (PL). Both PR and PL have been used to study the radiation damage by 5 KeV  $Ar^+$  and annealing behavior which is of interest in the context of surface cleaning of  $Mn_xCd_{1-x}Te$  prior to its use as an exactly lattice matched substrate for  $Cd_xHg_{1-x}Te$  epitaxy.  $Mn_xCd_{1-x}Te$  is substantially more resistant to radiation damage than CdTe, but exhibits a slower annealing kinetics. The primary point defects created by  $Ar^+$  bombardment are Cd Frenkel pairs. Investigations of the PC and PL spectra reveals that the conductivity at  $T < 100K$  is dominated by a very shallow acceptor, at 100K an electron trap of unknown origin is activated and at room temperature the electrical transport is determined by a deep acceptor. Since  $Mn_xCd_{1-x}Te$  may become of interest in the context of magnetically tunable lasers and photovoltaic applications further work on this material appears to be justified.

The remainder of this funding period will be devoted to the solution growth of  $Hg_{1-x}Cd_xTe$  on  $Cd_{1-z}Mn_zTe$  and related substrates. However, in future we will focus our attention to  $InAs_ySb_{1-y}$  and other potential substitutes for  $Cd_xHg_{1-x}Te$  that, in our opinion, is not the optimum material for far IR device applications. In all of the above alloy systems more or less restrictive solidus-liquidus separations exist with

associate striations in alloy composition under ground based conditions in the absence of a magnetic field. Therefore, we would like to continue our ground based research with a growth system suppressing convection by a magnetic field and plan for a future experiment a micro-g environment for more detailed evaluation of this aspect.

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Fluid Flow in Crystal Growth: Analysis of the Vertical  
Bridgman and Floating Zone Process

Massachusetts Institute of Technology  
Professor Robert A. Brown  
NSG-7645  
October 1, 1984 - September 31, 1985

Research is aimed at the fundamental understanding of the interactions of heat and mass transport and fluid mechanics in crystal growth from the melt, especially in how it pertains to experiments supported by the Microgravity Science and Applications program. Emphasis has been on studies of the vertical Bridgman system designed and constructed at MIT, on small-scale floating zone systems proposed for space flight, and on quantitative prediction of nonlinear transitions in melt/solid interface morphologies leading to the formation of cells and dendrites in directional solidification. Each research project applies new developments in the mathematical modelling of melt crystal growth and convection to problems of central interest for precision solidification of semiconductors and metals.

The analysis of vertical Bridgman growth has led to the complete numerical analysis of transport phenomena in directional solidification and to a detailed comparison of calculations to the GaGe growth experiments of Wang and Witt. Calculations show the central role of the ampoule material in determining the radial temperature gradients in the melt and thus the intensity of convection. Results indicate that diffusion-controlled growth can not be achieved on earth with conventional growth systems, but are feasible in microgravity. Addition of a stabilizing solute, such as silicon in germanium, allows diffusion-controlled growth on earth. Analysis is presently underway to understand the effect of externally applied magnetic fields on the structure of convection and on the transitions to time-periodic flow in an earth-bound systems. Full finite element calculations which incorporate the effect of the magnetic field in the model for vertical Bridgman growth are in progress.

A thermocapillary model of heat transfer and melt/gas interface shape in small-scale floating zones has been developed to predict zone shape and stability in experimental systems and is a precursor to a complete analysis of the interaction of fluid flow in the melt due to thermocapillary, buoyancy and crystal rotation with one shape and solute segregation. Calculations give the limits of validity of the cylindrical shape approximation often used in analyses. The thermal-capillary model is being extended to include convection in the melt caused by

buoyancy and surface-tension-gradients and to predict dopant segregation.

The analysis of nonlinear melt/solid interface morphologies in directional solidification has continued in an effort to predict theoretically the transitions between planar, cellular, and dendritic crystal growth. Using a combination of finite-element analysis and computer-implemented perturbation methods, we have predicted the details of the formation of large amplitude cells in two-dimensional interfaces and are presently analyzing for the onset of side-branching leading to dendritic structures. Calculations which account for the interactions of two cells show that low-amplitude time-periodic and chaotic states occur. These results may explain the failure of experiments aimed at observing the onset of a stable pattern. Two-dimensional solidification experiments with computer-controlled image processing have been initiated to test these hypotheses.

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## Crystal Growth of Device Quality GaAs in Space

Massachusetts Institute of Technology  
Professor Harry C. Gatos  
Dr. Jacek Lagowski  
NSG 7331 (NASA Contact: Dr. Alexander Tuyahov, HQ)  
April 1, 1985 - March 31, 1986

The ground-based program has been designed to guide and back up GaAs space growth programs. It combines three elements: crystal growth, device-related properties and characterization on a micro- and macro- scale. From that research two explicit GaAs space growth programs have branched out: (1) space growth of GaAs by electroepitaxy, which is the subject of a Joint Endeavor between NASA and Microgravity Research Associates, Inc.; and (2) space growth of GaAs using partially confined configuration, which is being carried out by the Electronic Materials Group at MIT. In the near future the program should evolve into its next logical stage whereby the ground-based research will be carried out simultaneously and in direct correlation with the space growth experiments.

The crystal growth study is designed to identify growth-property relationships critical for obtaining GaAs of improved device quality with an emphasis on characteristics which can be improved by materials processing in space. GaAs growth experiments involve: LEC growth with convection controlled by a magnetic field, Bridgman-type apparatus with precise control of the melt stoichiometry and the liquid phase electroepitaxy. The characterization and assessment of device-related properties is carried out utilizing the electronic characterization facility developed in the Electronic Materials Laboratory and the MIT Microelectronic Processing and Characterization facilities.

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## Morphological Stability and Fluid Dynamics of Vapor Crystal Growth

University of Utah  
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NSG-1534 (NASA Contact: Dr. A.L. Fripp, LaRC)  
1978 - 1986

This research is directed towards a fundamental understanding of the conditions under which crystals can retain morphological stability, i.e. shape stability of the advancing interface, during growth from vapors. Morphological stability (MS) is a necessary condition for the growth of homogeneous single crystals required for numerous device applications. For crystallization from melts, the MS concepts are well developed and are essentially based on heat and mass transfer conditions about the advancing interface. For crystallization from vapors, the MS requirements are more complex and not well understood. The added complexity arises from the fact that anisotropies in interfacial kinetics are typically stronger in crystallization from vapors than from melts. These pronounced anisotropies root in the distinctly lower atomic roughness of most vapor-solid interfaces.

The key insights obtained from the experimental and numerical work performed under this grant are: (1) with the incompressibility assumption (uncoupling of Navier-Stokes and energy equation), traditionally made in materials processing fluid dynamics, much of the essential physics is lost in simulations of vapor crystal growth processes; (2) even under zero-g conditions, the mere viscous interaction of diffusion fluxes with container walls leads to nonuniform concentration distributions (which, in turn, can act morphologically destabilizing); consequently (c) on earth, buoyancy-driven convection is always present in closed ampoule systems, irrespective of heating geometry and orientation of the transport flux with respect to "g". Utilizing these insights, the mass and heat transport prevailing about crystals during their growth from vapors are being investigated. Concentration fields in the vapor are studied by thermal deflection spectroscopy. Concurrently the evolution of the macroscopic morphology of the crystals are recorded by Moiree reflectometry.

The interfacial kinetics aspects of MS in vapor growth are being addressed through macroscopic studies of growth features and rates. Materials have been chosen, such as  $\text{CBr}_4$ , that, depending on the growth temperature, exhibit atomically rough or atomically smooth interfaces. Thus we will be able to correlate the existing, isotropic MS models with the anisotropic model as it emerges from our results. In this part of the work we have observed, for the first

time, surface roughening to occur as a precursor to a solid-solid transition. In addition we have expanded the traditional statistical treatment of atomic surface roughness to include the variation of bond strength at surfaces. The resulting model predictions agree well with experimental observation, in contrast to the constant bond models which fail to yield realistic predictions for vapor-solid systems.

This program is expected to provide sufficient insight into the diffusive-convective limitations of morphological stability in vapor crystal growth to ultimately warrant a purely diffusion-controlled bench mark experiment in space.

#### Publications

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Ming, N-B., Chen, J-S., and Rosenberger, F., "Surface Roughening as a Precursor to the Polymorphic Transition in  $CBr_4$ ," J. Cryst. Growth 69, 631 (1984).

Chen, J-S., Ming, N-B., and Rosenberger, F., "On the Atomic Roughness of Solid-Vapor Interfaces. Significance of Many-Body Interactions," J. Chem. Phys. 84, 2365 (1986).

Smutek, C., Bontoux, P., Roux, B., Schiroky, G. H., Hurford, A., DeVahl Davis, G., and Rosenberger, F., "Three-Dimensional Free Convection in Horizontal Cylinders. Numerical Solutions, Experimental Results and Analytical Approximations," Num. Heat Transfer 8, 613 (1985).

Bontoux, P., Roux, B., Schiroky, G. H., Markham, B., and Rosenberger, F., "Analytical and Two-Dimensional Approximations for Convection in the Midplane of a Horizontal Cylinder," Int. J. Heat Mass Transfer (accepted).

Experimental and Theoretical Analysis of Chemical Vapor  
Deposition with Prediction of Gravity Effects

Aerodyne Research, Inc.  
Dr. Charter D. Stinespring  
Professor Karl E. Spear, Penn State University  
NAS3-23934 (NASA Contact: Dr. G.J. Santoro, LeRC)  
September 29, 1983 - January 14, 1987

The overall objective of this program is to determine feasibility and general design of shuttle-borne experiments for assessing the effects of microgravity/gravity on CVD processes. To meet this objective, it is necessary to develop a quantitative understanding of gravitationally-induced transport processes in CVD systems and to determine the relationship between these transport processes and compositional and structural defects in CVD processes. Based on this knowledge, it will then be possible to recommend and interpret space-based microgravity experiments.

The approach in this program involves a combined experimental and theoretical study to characterize the effects of gravitationally-induced transport on atmospheric pressure silicon epitaxy by  $\text{SiH}_4$  pyrolysis. Experimentally, flow regimes in which free convective transport contributes to the CVD process will be identified, and, for these conditions, the flow and deposition process will be characterized. Specifically, this will include measurements of three dimensional temperature variations using in situ Rayleigh scattering, gas phase composition profiles using laser absorption and fluorescence techniques, and deposition rates and defect densities. Subsequently, the free convective transport contribution to the CVD process will be minimized and/or altered while leaving deposition chemistry unaltered, and the characterization will be repeated. Based on these analyses, the effects of gravitationally-induced transport on atmospheric pressure CVD will be assessed.

The theoretical component of the program seeks to develop and validate a combined fluid dynamics/chemistry model of CVD. To accomplish this, the modeling effort has been divided into two components. First, a two-dimensional fluid dynamics code for developing flow with variable fluid properties (parabolic problem) and a 2-D fluid dynamics code for fully-developed flow with variable fluid properties (elliptic problem) will be set up. These will then provide input for the second component which is a mass transport model with equilibrium and rate controlled chemistry coupled with the fluid dynamics. The predictions of this model will be tested against experimental observations.

## Publications

Wormhoudt, J., Stanton, A. C., and Silver, J., "Techniques for Characterization of Gas Phase Species in Plasma Etching and Vapor Deposition Processes," Proceedings of SPIE 452 (1983).

## Heat Flow and Segregation in Directional Solidification

Massachusetts Institute of Technology  
Professor August F. Witt  
NSG-7645

This research is composed of three major components:

- (1) development of interface morphology control for automated Bridgman growth of semiconductor systems. In attempts to optimize furnace design for crystal growth in a reduced gravity environment, an analytical approach to heat transfer was developed. It was thus found that charge confining crucibles diminish the ability to control the growth interface morphology through its position within the gradient zone. A heat pipe zone system for Bridgman growth, in reduced gravity environment, of crystal with diameters up to 16 mm was developed. Efforts are continuing to maximize the controllability of the growth interface morphology;
- (2) comparative analysis of segregation during crystal growth in a reduced gravity environment and in the presence of magnetic fields. For growth of Ga-doped germanium in the multipurpose (ASTP) furnace, it was found that the application of transverse magnetic fields (up to 36 kG) does not substantially increase the effective distribution coefficient, i.e. diffusion-controlled segregation observed in reduced gravity environment cannot be reached nor approached by magnetic field induced melt stabilization; and
- (3) consequences of seeding by melt-back in Bridgman growth under reduced gravity conditions. Experimental and theoretical studies established that steady state conditions cannot be reached during seeding, and that the growth interface temperature (interface location) during the initial stages of growth is a function of melt-back conditions. Numerical calculations were carried out for Ga-doped Ge and for HgCdTe.

### Publications

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Matthiesen, D. H., Wargo, M. J., and Witt, A. F., "Crystal Growth," in Opportunities for Academic Research in Low Gravity Environment, AIAA, in press.

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**2. SOLIDIFICATION OF METALS, ALLOYS  
AND COMPOSITES**

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Studies of Containerless Processing of Selected Nb-Based Alloys

Vanderbilt University  
Dr. Robert J. Bayuzick  
M. B. Robinson, MSFC  
NAG8-536 (NASA Contact: R.C. Black, MSFC)  
July 17, 1985 - July 16, 1987

Research is being conducted on the effect of containerless processing of alloys in a low-gravity environment. The primary goal is the better understanding of deep undercooling and its effect on microstructure and properties. The 100 meter drop tube at the Marshall Space Flight Center is being used to continue and extend work which has already been done to give a firm foundation of earth-based research.

Niobium-based binary alloys with tin, aluminum, germanium and silicon are being investigated for a number of reasons. First, the best drop tube experiments presently require alloys which solidify with large undercooling at or above 1700 K. Secondly, these four binary systems exhibit variation in their equilibrium behavior; a contrast of their undercooling behavior should contribute significantly to fundamental knowledge. Thirdly, each of these alloys is important technologically because of the existence, or potential existence, of a phase with the highest known superconduction transition temperatures. Finally, because of their refractory nature, work on these alloys provides a new dimension to materials processing in space; until now, experiments done and experiments planned have involved lower melting point metals and alloys.

Cornerstone of the proposed investigation is microstructural analysis. A number of complimentary techniques are being used to thoroughly characterize the effects of containerless processing. Optical microscopy, scanning electron microscopy with energy dispersive analysis and wavelength dispersive analysis of x-rays, transmission electron microscopy, and x-ray diffraction are all employed to obtain a complete description of structure. Measurement of bulk superconducting transition temperatures are being made by the self-inductance technique.

Publications

Bayuzick, R. J., Evans, N. D., Hofmeister, W. H., Johnson, K. R., and Robinson, M. B., "A Review of Long Drop Tubes as a Supplement/Alternative to Space Experiments," Adv. Space Res. 4, 85 (1984).

Hofmeister, W. H., Evans, N. D., Bayuzick, R. J., and Robinson, M. B., "Undercooling of Niobium-Germanium Alloys in a 100 Meter Drop Tube," in Rapidly Quenched Metals (S. Steeb and H. Warlimont, eds.), Elsevier, 1985, pp. 59-63.

Evans, N. D., Hofmeister, W. H., Bayuzick, R. J., and Robinson, M. B., "Solidification of Nb Alloys in Long Drop Tubes," Met. Trans. (in press).

Hofmeister, W. H., Evans, N. D., Bayuzick, R. J., and Robinson, M. B., "Microstructures of Niobium-Germanium Alloys Processed in Inert Gases in the 100 Meter Drop Tube," Met. Trans., 1986 (submitted).

## Measurement of High Temperature Thermophysical Properties

National Bureau of Standards  
Dr. D. W. Bonnell

This task has, as a primary aim, an ongoing evaluation of the experimental procedures needed in the collaborative interaction between Rice University (Rice) and the General Electric Company's Space Systems Division (GE). This effort focuses on technique development and the application of electromagnetic levitation to thermophysical property measurement, including determining heats of fusion, heat capacities, and enthalpy increment functions for extremely refractory materials. NBS involvement provides the background expertise in coupling techniques of electromagnetic levitation, auxiliary heating, such as electron beam, laser or other means, and drop calorimetry; supports design, operation and calibration of pyrometric temperature devices; and aids ongoing development of automation as prototypes for potential flight experiments.

Experimental studies are needed as high temperature heats of fusion, enthalpy functions and heat capacities ( $C_p$ ) are essentially all estimated for third row transition metals, and largely unknown for most other refractories with melting points above 2000 K. Theory has no predictive capability for these data, particularly at phase transitions, e.g., the change in  $C_p$ , solid to liquid, or the value of  $C_p$  near the melting point. There is not even a satisfactory explanation for why  $C_p(l)$  is apparently constant for refractory metals and generally much larger than the equipartition limit of  $3R$  ( $R = 8.314 \text{ J/g.mol}$ ). Accurate data should provide clues to improve parameters in existing models, or encourage the development of new models. As an example, current models for predicting alloy properties generally require good thermodynamic data of the constituents to obtain even approximate estimates.

Current efforts are concentrating on adapting the calorimetric data analysis codes to a microcomputer data acquisition system suitable for controlling the ground based system. The aim is to provide for immediate data reduction in ground-based experiments, and serve as the basis of a complete control and reporting scheme for the system needed for flight experiments.

Previous and ongoing major efforts include the development of techniques for imaging pyrometry at temperatures above 3500 K, a ground-based semiautomatic calorimeter system capable of handling liquid tungsten, and development of a two color imaging pyrometer system. This activity recently resulted in the first reported direct measurement

of the heat of fusion of liquid tungsten,  $53.0 \pm 2.3$  kJ/mol at 3695 K. This task and the collaborators' efforts represent an effort probing the boundary between ground based research and experimentation requiring the microgravity of space for success.

#### Publications

Bonnell, D. W., "Measurement of High Temperature Thermo-physical Properties," in NBS: Materials Measurements (J. R. Manning, ed.), NBSIR 85-3217, 1985.

Bonnell, D. W., Montgomery, R. L., Stephenson, B., Sundareswaran, P. C., and Margrave, J. L., "Levitation Calorimetry," in Specific Heat of Solids, Chapter 7 (A. Cezairliyan, ed.), McGraw-Hill, 1985.

## Dynamic Thermophysical Measurements in Space

National Bureau of Standards

Dr. Ared Cezairliyan

H-27954B (NASA Contact: R. Crouch, NASA HQ)

The objective of this research is to develop techniques for the dynamic measurement of selected thermophysical properties (e.g. heat of fusion, heat capacity, electrical resistivity) of high-melting-point electrically-conducting solids and liquids at temperatures above 2000 K in a microgravity environment.

The first phase of the research is to establish the geometrical stability of a specimen when heated rapidly to temperatures above its melting point in a microgravity environment. Work in this direction is underway. A test equipment package has been designed and constructed which permits rapid heating of the specimen to temperatures above its melting point and checking of the geometrical stability of the liquid specimen. This system consists of: removable specimen cartridge cells, a battery-bank power supply, a high-speed framing camera, a single-wavelength pyrometer, and electronic switching and control equipment. This system has been flown twice on board a KC-135 aircraft. The results suggested several refinements and modifications in the system, which are presently being made. Some additional work is needed in this direction to understand the behavior (geometrical stability) of the liquid specimen heated rapidly by the passage of a high current pulse through it, and as a result, optimize the specimen geometry and the operating conditions of the overall system. The second phase of the work is to add new measurement capabilities to the system and to demonstrate the applicability of the technique to performing definitive measurements of selected thermophysical properties of a refractory metal, such as niobium, in a microgravity environment.

Study on the Microstructures and Physical Properties of  
Materials Prepared in the Drop-Tube and Tower

University of Houston  
Dr. C. W. Chu  
NAG8-051  
November 1985 - November 1987

In collaboration with scientists from NASA/MSFC and the University of Alabama in Huntsville, several immiscible and near-eutectic material systems will be synthesized in the MSFC drop-tube and drop-tower facilities, or in the NASA KC-135 aircraft. The effects of microstructures induced by microgravity on the physical properties like electrical resistivity, magnetic strength, hardness, and corrosion resistance will be examined. Data obtained in these earth-based experiments will form the basis for experiments designed for Space Shuttle during the second phase of the research.

Metallurgical Study of Quench Rates Experienced by Drop-Tube  
Processed Samples

Battelle Memorial Institute  
Dr. E. W. Collings  
NAS8-36608 (NASA Contact: R. E. Black, MSFC)

Alloy samples are being processed in the NASA/MSFC 100-meter drop tube by levitation melting and dropping under various free-fall environmental and solidification conditions. Both in-flight solidification and splat quenching onto an inclined copper plate are being employed. Various metallurgical and physical properties of the products are being examined and compared with those of conventionally splat quenched materials.

The experimental work is being reinforced with calculations of free-fall cooling rates and of heat transfers to cold metal substrates. The potential levels of undercooling are being examined and modelled.



## Electrostatic Containerless Processing Technology

Jet Propulsion Laboratory  
Dr. Daniel D. Elleman  
Dr. W. K. Rhim  
In-Center

The long range objective of the task is the development of the science and technology base that is required to conduct contactless positioning and manipulation of high temperature materials using electrostatic and electro-phoretic forces.

The primary objectives to be addressed are experimental and theoretical investigations of: (1) the hybrid electrostatic-acoustic positioning module in both the 1-g laboratory environment and in the reduced gravity environment of KC-135 aircraft; (2) the high temperature focused radiator electric quadruple levitator; (3) the melting and solidification of metallic samples in the focused radiator furnace; and (4) the hot furnace using low density samples in one-g environment.

### Publications

Wang, T. G., Trinh, E. H., Rhim, W. K., Kerrisk, D., Barmatz, M., and Elleman, D. D., "Containerless Processing Technology at the Jet Propulsion Laboratory," Acta Astron. 11, 233 (1984).

Rhim, W. K., Collender, M., Hyson, M. T., Simms, W. T., and Elleman, D. D., "Development of Electrostatic Positioner for Space Material Processing," Rev. Sci. Instrum. 56, 307 (1985).

Naumann, R. J. and Elleman, D. D., "Containerless Processing Technology" in Materials Sciences in Space (R.J. Naumann and B. Feuerbacher, eds.), Springer-Verlag, 1986, pp. 294-310.

### Presentations

Rhim, W. K., "Electrostatic Levitation and its Application to Crystal Growth," to be presented at 9th Conference on Crystal Growth, June 1986.

## Studies of Model Immiscible Systems

Marshall Space Flight Center  
Dr. D. O. Frazier  
Dr. R. B. Owen  
B. R. Facemire  
W. K. Witherow  
Dr. W. F. Kaukler, UAH  
In-House  
October 1, 1982 - October 1, 1986

The objective of this research is to gain a fundamental understanding of the phase separation processes occurring during monotectic alloy production. The method by which this is accomplished depends significantly on the use of transparent model immiscibles cooling through a miscibility gap. Model immiscible systems contain a component which solidifies in a manner similar to that of metals. The reason for studying model systems rather than the metals themselves, is that the models are transparent and amenable to optical methods. Furthermore, it is probable that organic and aqueous systems lend themselves to ambient study more so than do metals.

One of the processes which separates the phases upon quenching into the coexistence region is sedimentation. Other effects are surface related and must be studied under microgravity conditions such as in the drop tower at MSFC, during ballistic aircraft trajectories, or on shuttle missions. In the laboratory, isotropic ratio adjustment may establish "neutral buoyancy" at a given temperature in some systems (e.g. deuterium substitution for hydrogen in succinonitrile-water).

Several experiments on the succinonitrile-water system provide compelling evidence that preferential wetting and thermal migration are important effects governing phase distribution in monotectic alloys. All of these observations are in cells that are either hydrophilic (normal pyrex glassware) or hydrophobic (silicon oil treated glassware). After quenching "neutrally-buoyant" succinonitrile-rich solutions, second phase droplet migration, due to interfacial tension gradients, drive the minority phase along the thermal gradient toward the hottest region which, in these experiments, is away from the crucible wall. If the minority phase preferentially wets the crucible wall, a minority phase layer can form adjacent to the solid surface and remain in the coldest part of the ingot. These surface effects are not as evident on the phase diagrams's water-rich side where the matrix-phase viscosity is three-fold less than that on the succinonitrile-rich side. We assume that in low viscosity matrices, convective effects dominate.

Techniques for obtaining fundamental information on surface effects include critical-point wetting, thermal migration, spinodal decomposition, Ostwald ripening, and nucleation and growth. Differential scanning calorimetry reveals strong clues as to the extent of the critical wetting region in the succinonitrile-water-pyrex system. Results of this phase of study is in preparation for publication.

#### Publications

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Witherow, W. K. and Facemire, B. R., "Optical Studies of a Binary Miscibility Gap System," J. Colloid & Interface Sci 104, 185-192 (1985).

Loo, B. H., Lee, Y. G., and Frazier, D. O., "Enhanced Raman Spectroscopic Study of the Adsorption of Dinitriles at the Copper-Aqueous Electrolyte Interface: Observation of Gauche and Trans Rotational Isomers of Succinonitrile on Copper Surfaces," J. Phys. Chem. 89, 4672-4676 (1985).

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## Gravitational Effect on Liquid Phase Sintering

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Professor Randall M. German  
Dr. E. P. Whelan, AMAX Materials Res. Center  
NAG3-744 (NASA Contact: Dr. G. Santoro, LeRC)  
October 1, 1986 - September 30, 1988

Experiments will be performed to determine the role of gravity in liquid phase sintering. Tungsten heavy alloys (tungsten-nickel-iron alloys) have been selected because of the large density difference between solid and liquid phases during sintering. Additionally, the investigators have considerable past experience in sintering these alloys (which are the subject of worldwide experimental study). The goal is to attain novel microstructures and shapes (without distortions) from eventual sintering under microgravity conditions. The current studies are measuring the coarsening, slumping and segregation that occur during normal sintering treatments. These studies will provide information on gravitational contributions to microstructural coarsening during liquid phase sintering. Additionally, the role of binder content, dihedral angle and liquid viscosity on dimensional stability during liquid phase sintering will be determined during the initial research.

The research is just beginning with a focus on the kinetics and mechanisms of grain growth. Previous theories have ignored the role that contiguity plays in coarsening. With increasing contiguity there is less solid-liquid interface, yet microstructural coarsening occurs rapidly. It is unknown what role gravity plays in the coarsening process. One theory proposes that new solid-solid contacts form due to liquid and solid migration under the influence of gravity. As a consequence, coalescence is proposed as a contributor to grain growth. Our early measurements will determine the effects of grain contacts on the coarsening kinetics, liquid migration and shape distortion during liquid phase sintering. These results will provide the basis for new concepts in liquid phase sintering, microstructural coarsening and help identify specific advantages from sintering under microgravity conditions.

Improvements in mechanical properties, shape complexity and dimensional control are envisioned from eventual low gravity sintering. These advances provide considerable interest in manufacturing these and other alloys in low gravity environments. Such attributes are of immediate industrial interest since liquid phase sintering is an ideal process for the fabrication of complex shapes from high performance materials. A current limitation is with low manufacturing yields and processing difficulties because of

shape distortion, microstructural coarsening and liquid migration induced by normal gravity. This research will identify the industrial manufacturing advantages from liquid phase sintering in low gravity environments.

The Role of Natural Convection on Crystallization from Vapor and Solution: A KC-135 and Laboratory Study

Desert Research Institute  
Dr. John Hallett  
NAS8-34605 (NASA Contact: V. Fogle, MSFC)  
February 2, 1983 - June 30, 1986

The objective of this research is to investigate the role of buoyancy induced convection in crystal growth and the differences which occur during growth in the absence of such convection under low-g.

Laboratory studies are being conducted on the role of convection in growth of: (a) ice vapor growth in presence of air (snow crystals); (b) ice growth from solution in presence of NaCl; and (c) sodium sulfate decahydrate from solution. Visualization of flow is achieved by Schlieren/Mach Zender optics. Enhanced flow can be achieved by a wind tunnel or moving the crystal during growth. The system is uniformly supercooled so that the crystal grows into a well defined environment. Of particular interest is the facet - dendrite transition which occurs at a critical supersaturation/supercooling, which is dependent on the ventilation velocity. This transition changes to lower supercooling/supersaturation with the absence of convection in low-g in KC-135 flights.

Presentations

Hallett, J., "Ventilation and Growth Form of Crystals," presented at OJI International Seminar on Morphology and Growth Unit of Crystals, Japan, August 1985.

Hallett, J., "Disc Growth of Ice Crystals from the Vapor," presented at OJI International Seminar on Morphology and Growth Unit of Crystals, Japan, August 1985.

The Development and Prevention of Channel Segregation During Alloy Solidification

Michigan Technological University

Dr. Angus Hellawell

NAG3-560 (NASA Contact: Dr. R. L. Dreshfield, LeRC)

July 25, 1986 - July 24, 1988

The objective of this research is to better understand how channels of high solute concentration develop within the partly solidified region of an ingot. The problems are concerned with how/where such channels originate; how, once established, they propagate with given dimensions and spacings, and, what methods can be used to avoid their formation.

An experimental program is underway with aqueous salt solutions and with lead-tin alloys. The principal geometrical arrangement is one having a base chill with upward growth, antiparallel to gravity. Studies of the transparent system have been to gain qualitative insight about the processes; studies of the metal system are designed to relate the channel formation and elimination to the permeability of the dendritic mesh, by changing alloy composition.

Publications

Sample, A. K. and Hellawell, A., "The Use of Analog Castings to Study Channel Segregation," in Modelling of Casting and Welding Processes, AIME, 1984, pp. 119-125.

Sample, A. K. and Hellawell, A., "The Mechanisms of Formation and Prevention of Channel Segregation During Alloy Solidification," Met. Trans. A15, 2163 (1984).

Presentations

Hellawell, A. and Sarazin, J. R., "Fundamentals of Solidification," presented at TMS-AIME Fall Meeting, Toronto, 1985.

Whisker Growth Studies Under Conditions Which Resemble Those Available on an Orbiting Space Laboratory

George Washington University  
Dr. Herman H. Hobbs  
NAG3-642 (NASA Contact: L. Westfall, LeRC)  
June 10, 1985 - June 10, 1986

The objectives of this research task are: (1) the determination of advantages, disadvantages, and special circumstances attendant upon annulment of earth's surface gravity during nucleation and growth of metal crystals (especially whiskers), and (2) determination of possible analytical (or practical) uses for electric currents which are a concomitant of the procedure used to levitate the growing whiskers.

Whiskers are grown by the chemical reduction of metal halides in the presence of applied electric fields. The applied fields have a number of effects including levitating forces on the whiskers. The fields can partially (or fully) support the growing whiskers and aids in growth by preventing the young whisker nuclei from falling over into the molten metal halide growth substance. This process is accompanied by an electric current which could be highly useful if current studies yield an understanding of its origin. To further mimic orbital conditions steps are being taken to suppress the convection currents which are usually attendant upon this growth process, and to perform all growth experiments in a vacuum chamber which will permit use of low and quickly variable partial pressures.



## Control of Solidification Structure of Germanium-Silicon Solutions

Auburn University  
Dr. Wartan A. Jemian  
NAG8-491 (NASA Contact: R. E. Black, MSFC)

The objective of this research is to develop information on the relation of solidification process variables to the form and quality of the resulting materials. It is already known that a variety of morphologies, including planar and cellular growth crystals, several types of dendrites, whiskers and amorphous mixtures are formed in this system. The most desirable result is a homogeneous, planar growth crystal with low defect density.

The initial phase of the research involved small sample studies conducted at Auburn University. The results contributed information to the solidification structure-process control map, which shows the ranges of the solidification variables associated with different growth forms. This work will be extended using the drop tube research facility at MSFC. The second phase of the research is to establish specific process boundaries and identify growth mechanisms. The final phase of the research will be the study of physical characteristics of specific samples.

This research approaches the problem of producing device quality materials in a unique and innovative manner. It is directed towards finding a method to use the microgravity environment for its demonstrated advantages in achieving high perfection solidification structures even in alloy systems prone to constitutional defect generation. The liquid sample is released while attached to an oriented seed crystal. The seed is sufficiently large to act as the major heat sink, thus accelerating growth. Conduction into the seed is much more effective than radiation cooling. Since solidification takes place in a microgravity environment, the range of conditions for planar growth will be extended, beyond those under the usual ground-based laboratory conditions.

## Verification of Critical Point Wetting Theory Using Microgravity

University of Alabama in Huntsville  
Dr. William F. Kaukler  
NAG8-51 (NASA Contact: R.E. Black, MSFC)  
January 18, 1985 - January 17, 1987

Wetting and fluid behavior near the critical temperature can be better observed in a microgravity environment, which could be provided by the Drop Tower at MSFC. Whether the two fluid phases in question are immiscible liquids or a liquid and its vapor, the problem of hydrostatic pressure caused by the density differences between the two phases will interfere with measurements of contact angle or interface curvature if the experiments are performed in a one-g environment. Microgravity will be used as a tool to verify the critical point wetting (CPW) theory of John Cahn.

There are tenets of the CPW theory that have not been tested. The claim was made that the theory is generally applicable to all systems where two fluid phases undergo a critical phase transition in contact with a third phase. There is some evidence of the critical point wetting phenomena in immiscible systems. There is no evidence that a single component, liquid-vapor-container type system will experience the CPW transition. Testing the theory on a single component in order to provide the generality of the theory is one objective of the research.

Processes over which CPW has some active controlling part are found in several aspects of materials processing. Surface tension forces and concentration gradients found in some systems become significant concerns in space because the relatively powerful masking effect of gravity-induced convection is removed. The forces involved with CPW are weak, but may in fact dominate in a low-g environment. A space environment provides advantages when processing materials which require fluid phases. This includes solidification and crystal growth. During the solidification of monotectics, it is expected that CPW has an influence upon the microstructure of the final product. Cryogenic liquid gases like hydrogen and oxygen are stored in containers at near atmospheric pressure. The critical temperature and pressure of these liquids are both very low. It is conceivable that in a space environment, at low-g, CPW phenomena may be observed in these fields. This of course is of interest to the propellant engineers for rocket engines. Verification of the CPW theory with single component systems is therefore needed.

Four related but different experiments will be performed. They test the critical point wetting theory of

John Cahn and can be used to measure interfacial free energies between two fluid phases near their critical temperature, and of the container-fluid interfacial energy as well. Two system types will be experimented with, single component, vapor-liquid systems and two-component, liquid-liquid systems. The inter-fluid interface shape will be measured in order to obtain the Bond number and eventually derive the approximate interfacial free energy. The contact angle the inter-fluid interface makes with the container will be measured, and the period of oscillation of the inter-fluid interface will be measured in order to directly obtain the interfacial free energy of the oscillating interface. Experiments will be repeated for a series of temperatures in order to find the CPW transition temperatures for each system.

Finding the  $T_w$  for single component and for binary systems is the main objective of the experiments and will show the generality of the theory empirically. Once this is done, the theory can be applied and tested on a system selected specifically to test the connection between the CPW theory and the behavior of superfluid-He.

Structure of Nickel and Iron Aluminides Prepared by Rapid Solidification and Undercooling

North Carolina State University  
Dr. Carl C. Koch  
NAG8-475 (NASA Contact: R.E. Black, MSFC)  
August 1, 1984 - January 31, 1986

The objective of this investigation is to obtain a basic understanding of the complex solidification structures found in the nickel and iron aluminides by combining rapid solidification studies in foils and ribbons with undercooling studies in the MSFC drop tube.

Metastable microstructures, as revealed by transmission electron microscopy, have been obtained in Ni<sub>3</sub>Al (with B) samples solidified in the MSFC drop tube. Anti-phase domain boundaries, which are not seen in conventionally cast alloys were observed. This indicates undercooling has occurred. Parallel studies of rapidly solidified alloys produced in the arc-hammer at NCSU have been carried out. After a new detector is installed on the MSFC drop tube, quantitative measurements of undercooling will be made and comparisons between the solidification behavior of the rapidly solidified and undercooled samples will be made.

Publications

Koch, C. C., "A Review of Rapid Solidification Studies of Intermetallic Compounds," in High-Temperature Ordered Intermetallic Alloys (C.C. Koch, C.T. Liu, and N.S. Stoloff, eds.), MRS, 1985, pp. 397-410.

Koch, C. C., "Testing Techniques for Mechanical Characterization of Rapidly Solidified Materials," in Mechanical Behavior of Rapidly Solidified Materials (S.M.L. Sastry and B.A. MacDonald, eds.), TMS, 1986, pp. 41-57.

## Metallic Glass Research in Space

Jet Propulsion Laboratory

Dr. Mark C. Lee

Dr. Taylor G. Wang

NAS7-918

October 1, 1985 - September 30, 1987

The objective of this research is to develop a space experiment to acquire thermodynamic properties of bulk metallic glasses over the entire undercooling region, with emphasis on the temperature region inaccessible by terrestrial techniques.

Ground-based precursory experiments will be designed and performed in such a manner that all the critical parameters for the space experiment will be defined and validated. A logical approach to achieve this goal should include the following ground-based tasks: (1) development of a novel contactless calorimetry technique for specific heat measurements over the entire undercooling region of a bulk metallic glass sample; (2) measurements of specific heats and crystallization kinetics to precisely define the optimal candidate systems for the space experiment; (3) conceptualization of the data analysis technique for the space experiment; and (4) feasibility study of the metallic glass space experiment module.

### Publications

Annamalai, P., Lee, M. C., Crawley, R. L., and Downs, R. L., "Polyvinyl Alcohol Coating Polystyrene Inertial Confinement Fusion Targets," J. Vac. Sci. Tech. 3, 1204 (1985).

Lee, M. C., "Formation of Metallic and Metallic-Glass Hollow Spheres and Their Solidification Characteristics," in Rapidly Quenched Metals (S. Steeb and H. Warlimont, eds.), Elsevier, 1985, pp. 119-122.

Lee, M. C., "Hybrid Contactless Materials Processing Apparatus," NASA Tech Brief, Spring 1985.

Lee, M. C., "Blowing Polymer Bubbles in an Acoustic Levitator," NASA Tech Brief, Summer 1985, p. 100.

Barmatz, M. B. and Lee, M. C., "Acoustic Pressure Measurement," NASA Tech Brief, Winter 1985, p. 46.

Lee, M. C., Kendall, J. M., Bahrami, P., and Wang, T. G., "Sensational Spherical Shells," Aerospace America, 1986 (in press).

## Containerless Processing of Undercooled Melts

University of Wisconsin, Madison  
Professor John H. Perepezko  
NAG3-436 (NASA Contact: T. Glasgow, LeRC)  
September 1, 1983 - August 31, 1986

A central objective of the research is to extend the understanding of the physical mechanisms controlling liquid undercooling and to apply this knowledge to microgravity solidification processing. An assessment of the undercooling potential of containerless processing is being conducted on powder samples in a laboratory-scale drop tube apparatus. The nucleation and crystal growth behavior in undercooled liquids are under study in several iron and nickel-base systems. The processing parameters to be examined include melt superheat, droplet size and particle statistics and droplet surface coating. The solidification behavior is determined by thermal analysis and by structural and metallographic characterization.

The availability of thermal history information such as the crystallization onset temperature, can be essential for the proper interpretation of a solidification microstructure following drop tube processing. During the current research period a number of coordinated efforts have been directed towards obtaining thermal history information. In one approach the InSb-Sb eutectic microstructure developed during drop tube processing has been compared to that produced at known undercoolings as measured by differential thermal analysis. The experimentally measured eutectic spacing-undercooling relation has been used to establish an undercooling for containerlessly processed samples up to  $0.2 T_E$ . An alternate method is based upon the use of structural transitions to judge undercooling levels. In a Ni-53 w/o Nb alloy, a crystal-glass transition that develops in fine droplets ( $< 30 \mu$ ) indicates a substantial undercooling in excess of 500 C. The droplet fraction exhibiting this transition is also sensitive to drop tube gas environment which is being used to examine surface coating effects. A third approach involves modeling the heat transfer during containerless processing to calculate the thermal history and testing the model with microstructural observations.

The continuing program will emphasize the development of laboratory scale drop tube studies during the next year. The experience will be extended to high temperature systems including further work on Ni-Nb alloys and studies on structural transitions in Fe-Ni and Fe-Mn alloys. Further attention will be focused on monitoring the thermal history of drop samples by microstructure study and the development of direct measurement methods. Solidification kinetics

measurements will also be included into the evaluation of the undercooling capability of containerless processing.

#### Publications

Graves, J. A., "Undercooling and Solidification Behavior in the InSb-Sb System," NASA CR-175013, 1985.

Boettinger, W. J. and Perepezko, J. H., "Fundamentals of Rapid Solidification," in Rapidly Solidified Crystallizing Alloys (C. Adam, S. Das, and B. Kear, eds.), AIME, (in press).

Graves, J. A. and Perepezko, J. H., "Undercooling and Crystallization Behavior of Antimony Droplets," J. Mat. Sci., 1985 (in press).

#### Presentations

Perepezko, J. H., "Containerless Processing of Undercooled Melts," presented at Gordon Research Conference on Microgravity Science, August 1985.

Graves, J. A. and Perepezko, J. H., "Solidification of a Normal-Type InSb-Sb Eutectic in Undercooled Droplets," presented at TMS-AIME Fall Meeting, Toronto, October 1985.

## Directional Solidification of Bi-Mn and Pb-Bi in an Applied Magnetic Field

Grumman Corporate Research Center  
Dr. R. G. Pirich  
J. L. DeCarlo  
NAS8-36106 (NASA Contact: F. Reeves, MSFC)  
July 15, 1984 - October 15, 1985

Selected eutectic, off-eutectic Bi-Mn and peritectic Pb-Bi compositions have been directionally solidified in applied magnetic fields. The objective of the research is to determine what effect the magnetic field has during solidification on thermal and solutal instabilities in the melt by characterizing the resulting growth structure and electronic properties. Off-eutectic Bi-Mn and peritectic Pb-Bi were grown parallel and antiparallel to the gravity direction with and without transverse applied magnetic fields of up to 3kG. An eutectic (0.72 w/o Mn) Bi-Mn sample was grown antiparallel to gravity in a longitudinal magnetic field that varied linearly from 0-40kG and back to 0kG over a 200 minute period.

Both Mn-rich (0.94 w/o Mn and 1.65 w/o Mn) and Bi-rich (0.26 w/o Mn and 0.58 w/o Mn) samples were chosen for growth so that in combination with parallel/antiparallel growth orientations, they provided various stable/unstable solutal conditions. Thermal instability effects were also studied in connection with parallel gravity vector growth. Growth rates were 0.7 cm/h and 10 cm/h for off-eutectic Bi-Mn and 2 cm/h for peritectic Pb-Bi. Eutectic Bi/Mn was grown at 0.5 cm/h antiparallel to gravity as previous studies have shown that no cooperative growth occurs in this velocity regime for 1-g conditions with no applied magnetic field. Initial results from this experiment show that cooperative growth did occur at field values of roughly 16-18 kG. Also, preliminary examination of the off-eutectic work shows the magnetic field apparently reduced mixing effects for the 0.26 w/o Mn bulk composition samples grown in solutally unstable orientations. Analysis of the Mn concentration versus fraction solidified for the 0.58 w/o Mn bulk composition samples indicate that application of the magnetic field increased the macrosegregation compared to samples grown without the magnetic field. Of the Mn-rich samples (0.94 w/o Mn, 1.65 w/o Mn), Mn concentration as a function of fraction solidified indicated that growth parallel to gravity was the only stable condition with or without a magnetic field. Growth in the antiparallel direction resulted in an inverse macrosegregation distribution apparently from Stokes' flow of MnBi dendrites. The 1.65 w/o Mn bulk composition samples and the peritectic Pb-Bi samples are still being studied.



Future plans are to investigate the effects of applied magnetic fields on directional solidification of CdTe and CdSe<sub>1-x</sub>Te<sub>x</sub> alloys and peritectic Pb-Bi alloys. Further growth studies at high fields (40 kG) at the Francis Bitter National Magnetic Laboratory will also be conducted.

## The Role of Gravity on Macrosegregation in Alloys

University of Arizona  
Professor D. R. Poirier  
Dr. C. F. Chen  
NAG3-723 (NASA Contact: Dr. R.L. Dreshfield, LeRC)  
1986 - 1989

The objective of this research is to conduct fundamental research on the phenomenon of macrosegregation during solidification of alloys. The major thrusts of the research are to develop a computer code to simulate macrosegregation phenomena and to consolidate the findings of several shuttle experiments dealing with macrosegregation. In addition to drawing on the existing and new knowledge of macrosegregation and solidification phenomena, experimental results generated by complementary programs will be analyzed quantitatively.

The research includes experiments designed to obtain physical data which are crucial to successfully simulating solidification. Specifically, partition ratios, freezing points, and densities of the liquid-and solid-phases will have to be determined for the multicomponent alloy. These data are available for the binary alloys selected for the study (Pb-15 Wt pct. Sn and Sn-15 wt. pct. In). The permeability for the flow of interdendritic liquid in dendritic networks of Pb-Sn alloys is being determined. The research will include experiments to determine the permeability in Sn-15 Wt. pct.

The computer model will be applied to the analysis of macrosegregation in ingots of circular symmetry which are rotated around their axes and ultimately to ingots which are rotated in a more complex manner. This analytical experimental analysis would emphasize the role of a centrifugal body force on the convection field in the presence of a gravity and in the microgravity environment. Multicomponent alloys will be studied primarily to involve industry and pave the way to commercial utilization of the practical aspects of process modeling which will result from the overall program.

## Graphite Formation in Cast Iron

University of Alabama  
Professor Doru M. Stefanescu  
NAG8-469 (NASA Contact: R. Mixon, MSFC)  
June 1985 - July 1987

The objectives of this research are: (1) to better understand the solidification mechanics of cast-iron and similar type alloys using directional solidification experiments in microgravity; (2) to determine the contribution of gravity dependent effects on the final microstructure and properties of the alloys; (3) to investigate unique microstructures that may be obtainable by processing of alloys in a microgravity environment; and (4) to make the results of the study available for application in improving terrestrial casting techniques.

Results of experiments performed in the first part of the project have been reported in the references below. For Fe-C-Si alloys solidifying with stable eutectic (with either lamellar or spheroidal graphite), it was concluded that solidification under low-g results in a decreased number of eutectic grains, which could be attributed to a decrease in nucleation because of the change in the wetting properties of substrates occurring in low-g processing. Also, low-g processing resulted in an increase in the secondary dendrite arm spacing, with a subsequent decrease during high-g zones. Further it has been shown that buoyancy-driven graphite phase segregation can be prevented during low-g processing. In the metastable Fe-C eutectic alloys, a refinement of interlamellar spacing has been observed during low-g processing.

In the second part of the research partially reported in reference 3, it was found for two different systems, Fe-C-Si and Fe-C-V, that primary particles (spheroidal graphite and vanadium carbide) tend to reach larger sizes when solidifying in the low-g zone as compared with the high-g zone, during parabolic flights. Calculations have shown that under the described experimental conditions, particles were either entrapped or have floated, which explains the rather complex microstructures obtained.

Further experimental work will be conducted at lower solidification rates to check the validity of Chernov's theoretical approach on particle pushing/entrapment.

## Publications

Hendrix, J. C., Curreri, P. A., and Stefanescu, D. M., "Directional Solidification of Flake and Spheroidal Graphite Cast Iron in Low and Normal Gravity Environment," AFS Trans. 99, 435 (1984).

Stefanescu, D. M., Curreri, P. A., and Fiske, M. R., "Microstructural Variations Induced by Gravity Level During Directional Solidification of Near-Eutectic Iron-Carbon Type Alloys," Met. Trans. 17A, 1121 (1986).

Stefanescu, D. M., Fiske, M. R., and Curreri, P. A., "Behavior of Insoluble Particles During Parabolic Flights Solidification Processing of Fe-C-Si and Fe-C-V Alloys," in Proceedings of 18th SAMPE Technical Conference (in press).

A Systematic Study of Density Sensitive Micro-Structures in Some Monotectic Alloys

University of Alabama  
Dr. Doru M. Stefanescu  
NAS8-36193 (NASA Contact: P.A. Curreri, MSFC)  
November 1984 - January 1986

Monotectic alloys in the Cu-Pb system were directionally solidified in a Bridgman-type furnace on ground and during parabolic flights on the KC-135 aircraft. Solidification rates of 0.15 to 10 mm/min. were used, as well as lead contents of 8-25%Pb. At low Pb contents (10-30%) primary Cu dendrites were found as predicted by Livingston and Cline. At about 30 to 45%Pb a rod composite structure was seen, while at more than 45%Pb a droplet-type structure was evident. Decreasing the solidification rate at 0.15 mm/min. some new types of structures were identified: an irregular droplet-type structure and a band-structure. The change from droplets to dendrites is accompanied by an increase in the average lead content of the sample, indicating that lead was incorporated at the interface.

From ground and flight results, it is proposed that a lead droplet structure can also solidify at low lead contents and low solidification rates. In addition, it is suggested that the banding seen at lower rates and high lead contents results from lead being pushed by a very stable interface and then suddenly incorporated by it. The stability of this interface breaks down with increasing rate and becomes the droplet structure identified by Livingston and Cline.

A new solidification rate - lead content - structure diagram was produced.

## Theoretical Studies of the Surface Tension of Liquid Metals

Ohio State University  
Professor David G. Stroud  
Dr. Wei-Heng Shih  
Dr. Malcom Grimson  
NAG8-483 (NASA Contact: T.P. Crabb, MSFC)  
July 1, 1984 - July 1, 1987

The objectives of this research are to understand the surface tension and bulk properties of liquid metals from a knowledge of statistical mechanics of liquids combined with the electronic theory of metals. Both liquid-vapor and liquid-solid surface tensions are of interest. The liquid-vapor surface tension of several liquid metals is being studied theoretically as a function of temperature and impurity concentration in order to understand surface-tension-driven flows in a microgravity environment. The liquid-solid surface tension is being calculated for several liquid metals, and for liquid semiconductors, in order to understand the parameters which enter nucleation theory. Thermodynamic properties (compressibility, specific heat) of bulk liquid metals are also being calculated, in order to make contact with experimental programs.

Liquid-vapor and solid-liquid surface tensions are being studied by two different methods. The first is a density-functional method which permits the calculation of the free energy of an inhomogeneous liquid metal in terms of that of a homogeneous metal; this method has already yielded good results for liquid-vapor surface tensions of several liquid metals and liquid metal alloys. Preliminary results for liquid-solid surface tensions are also promising. The second method is a semi-empirical lattice gas approach which allows the surface properties of liquid metals and alloys to be expressed conveniently in terms of bulk properties. Both methods show that, in certain ranges of concentration, alloys can have negative surface entropy, i.e. a positive temperature derivative of surface tension. Bulk thermodynamic properties are being studied by pseudo-potential perturbation theory, combined with a thermodynamic variational method, for the simple (i.e. nearly free electron) metals and for liquid elemental semiconductors. A program to calculate these properties using a newly developed molecular dynamics scheme (which will allow computation of dynamical properties such as diffusivity) is just getting underway.

## Publications

Shih, W. H. and Stroud, D., "Theoretical Study of Miscibility and Glass-Formed Trends in Mixtures of Polystyrene Spheres," J. Chem. Phys. **80**, 4429 (1984).

Stroud, D. and Grimson, M. J., "Density-Functional Calculation of the Surface Tension of Liquid Al and Na," J. Non-cryst. Solids **68**, 231 (1985).

Shih, W. H. and Stroud, D., "Theory of the Surface Tension of Liquid Metal Alloys," Phys. Rev. **B32**, 804 (1985).

Shih, W. H. and Stroud, D., "Thermodynamic Properties of Liquid Si and Ge," Phys. Rev. **B31**, 3715 (1985).

Shih, W. H., Ebner, C., and Stroud, D., "Potts Lattice Gas Model for the Solid-Liquid Interfacial Tensions of Simple Fluids," Phys. Rev. B (in press).

Shih, W. H. and Stroud, D., "Two-Component Lattice Gas Model for Surface Segregation in Liquid Alloys," Phys. Rev. B (in press).

Shih, W. H., Hirth, J. P., and Stroud, D., "Twin Boundary Energy and Entropy of Simple Metals: A Constant Pressure Monte Carlo Calculation for Al," Phys. Rev. B (in press).

## Solidification Fundamentals

NASA Lewis Research Center

Dr. S. N. Tewari

Dr. A. Chopra

In-House (NASA Contact: Dr. Hugh Gray, LeRC)

January 1984 - continuing task

Dr. S. N. Tewari, supported by the National Research Council, has completed the initial phase of the quantitative microstructural analysis of directionally solidified alloy samples. He has compared his results with several theoretical models for dendritic growth in a positive temperature gradient (DS). These models are based on the work of Burden and Hunt, as well as Laxmanan and Trivedi. These models have been quantitatively compared with the dendrite tip radius, dendrite tip temperature, and primary arm spacing data available in literature. The predictions due to both Laxmanan (minimum undercooling approach) and Trivedi (marginal stability approach) show very good fit for the alloys examined: succinonitrile-acetone, succinonitrilealcohol, aluminum-copper, and lead-palladium. Pb-3% Pd and Pb-8% Au alloy specimens, directionally solidified and quenched, were examined for their composition profiles, both within the interdendritic region and ahead into the melt. Dendrite tip radius measurements were carried out by a careful sectioning technique. To distinguish between the proposed models, directional solidification experiments must be carried out at growth rates significantly lower than previously attempted, and in the absence of experimental wall effects and convection. Dr. Tewari has coordinated the overall design and fabrication of an innovative directional solidification furnace which will be used later in the Microgravity Materials Science Laboratory to conduct the required critical experiments.

Dr. A. Chopra, supported by the National Research Council, has participated in the design and fabrication of an isothermal dendrite growth apparatus (IDGA). This is a prototype of Professor Martin Glicksman's flight experiment apparatus. It is now ready for checkout and ground-based experimentation in the Microgravity Materials Science Laboratory. Dr. Chopra has recently determined the phase diagram for the SCN-oil red "alloy" system in preparation for planned research experiments on dendrite growth mechanisms using the IDGA.



## Publications

Tewari, S. N., "Effect of Undercooling on the Microstructure of Ni-35%Mo (Eutectic) and Ni-38%Mo (Hypereutectic) Alloys," Met. Trans., 1985 (accepted).

Jayaraman, N., Tewari, S. N., Hemker, K. J., and Glasgow, T. K., "Microstructures in Rapidly Solidified Ni-Mo Alloys," in Proceedings of ASM International Conference on Rapidly Solidified Materials, 1985 (accepted).

Tewari, S. N., "Undercooling in Ni-31%Mo Alloy," J. Cryst. Growth, 1985 (submitted).

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Jayaraman, N., Tewari, S. N., Hemker, K. J., and Glasgow, T. K., "Microstructure in Rapidly Solidified Ni-Mo Alloys," presented at ASM International Conference on Rapidly Solidified Materials, San Diego, February 1986.

Tewari, S. N. and Glasgow, T. K., "Undercooled and Rapidly Quenched Ni-Mo Alloys," presented at AIME Symposium on Undercooled Alloy Phases, New Orleans, March 1986.

## Containerless Studies of Nucleation and Undercooling

Jet Propulsion Laboratory  
Dr. Eugene H. Trinh  
NAS7-918

The long term research goals are to perform experiments to determine the achievable limits of undercooling using acoustic levitation, to study the characteristics of heterogeneous nucleation of levitated samples, and to measure the physical properties of significantly undercooled melts. Specially designed ultrasonic levitators operating in ground based laboratories as well as in the KC-135 NASA aircraft are to be used to investigate 0.1 to 3 mm specimens of pure metals and alloys (Ga, In, Sn, Al-In,...) as well as glass-forming organic compounds (O-Terphenyl, low melting glasses). Non-invasive measurement techniques for the surface tension, viscosity, density, sound velocity, and perhaps specific heat, are to be developed and refined to probe the physical state of undercooled levitated melts.

Initial melting and undercooling of Ga, In, and Sn have been performed in an inert atmosphere of Argon at one Bar, and strongly underscore the importance of the problem of contamination by impurities and oxide layers coating the surface of a levitated melt. One of the major goals has thus become the redesign of levitators in order to minimize the influence of such foreign substances and to control the environment in which the samples are levitated. Undercooling and freezing of melts in a levitator operating under the full effect of the earth's gravitational field have revealed the importance of the ultrasonic field on the large scale morphology of the resulting solid sample, thus motivating a detailed study of the interaction between the crystallization and fluid dynamic phenomena dominated by the high intensity acoustic field. Finally, measurement techniques based on the physical acoustic principles are being refined for the determination of the density, surface tension, viscosity, and refraction index of undercooled melts.

### Publications

Marston, P. L. and Trinh, E. H., "Hyperbolic Umbilic Diffraction Catastrophe and Rainbow Scattering from Spheroidal Drops," Nature 312, 529 (1985).

Trinh, E. H., "Compact Acoustic Levitation Device for Studies in Fluid Dynamics and Material Science in the Laboratory and Microgravity," Rev. Sci. Instrum. 56, 2059 (1985).

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Trinh, E. H., Robey, J. L., Jacobi, N., and Wang, T. G., "Dual Temperature Acoustic Levitation and Sample Transport Apparatus," J. Acoust. Soc. Am. 79, 604 (1986).

Trinh, E. H., "Application of Acoustic Levitation to the Investigation of Melting and Freezing Phenomena," J. Acoust. Soc. Am. 79, 55 (1986).

## Ostwald Ripening of Solid-Liquid Mixtures

National Bureau of Standards

Dr. P. W. Voorhees

Dr. J. R. Manning

S. C. Hardy

H-85025B (NASA Contact: F. Reeves, MSFC)

The objective of this program is to use the special conditions provided by space flight to study the role of the volume fraction of coarsening phase and convection during Ostwald ripening. Information concerning this process will be important for predicting and controlling the processing of a variety of materials containing dispersed phases.

A particularly ideal system to use in these experiments is a mixture consisting of solid particles in a liquid. Since the coarsening rate in such a system is comparatively fast and in a properly chosen system the solid particles are nearly spherical, the experiments can serve as a careful test of theory, assuming that the solid particles remain fixed in space. However, under terrestrial conditions at low volume fractions solid, buoyancy driven convection of the solid particles is prevalent and thus the experiments do not satisfy the theoretical requirements of fixed spatial locations. To eliminate this problem the experiments will be performed in the reduced gravity environment of space. As a result of this reduced convection, it is expected that the first data on the coarsening kinetics of low volume fraction two-phase mixtures which is directly comparable to theory will be obtained.

In initial work, we have succeeded in producing mixtures consisting of spherical solid particles in liquid in two systems which are promising candidates for the space flight experiment. Quantitative characterization of these mixtures is currently underway.

## Spherical Shell Technology

Jet Propulsion Laboratory  
Dr. Taylor G. Wang  
In-Center  
October 1978 - continuing task

The primary objective of this task is to investigate the science and technology pertinent to the production of spherical shells and to study the effect of gravitation on the formation of spherical shells both in the laboratory and in a weightless environment. The technology base being developed includes, but is not limited to, the fluid dynamics of viscous media, metallic, and amorphous materials. This technology is being applied to the process of shell fabrication; for rendering the shell spherically symmetrical; to the sintering of the shell into a composite matrix material; to the development of the production of a novel high-strength, low-weight material for bonding of the spheres; and to the development of techniques needed for the encapsulation of various materials within the spherical shell such as phase-change materials for heat regeneration.

We have developed a unique fluid-dynamic approach for the production of spherical shell from a wide range of materials at a high rate of speed and with minimal cost. We have formed shells of metal, plastic, and glass. Size ranges from 0.4 to 5.0 mm and we have determined gravitational effects on shell concentricity. This technology has already attracted many industrial sponsors. Currently, two companies have negotiated with NASA on commercializing the product.

A theoretical model for the annular jet instability has been developed in which the liquid layer enclosing the gaseous stream in the jet is modeled as a membrane with no thickness but finite mass/area which moves under the influences of its own surface tension, inertia, and the gaseous pressure. It should be noted that the hollow jet instability is a phenomenon completely different from the more familiar Rayleigh instability of a simple jet. The methodology of converting a free surface flow problem into a one-dimensional one using a thin sheet model that can be handled more easily is an innovative contribution to the field of theoretical nonlinear physics.

We have derived a microscopic model of first-order phase changes in a none component system with spherically symmetric, two-body potentials. Included is a microscopic model for the driving forces inducing the phase change, cooperative model for homogeneous nucleation, and a possible solution to the "fcc-hcp" problem in rare gases.

## Publications

Lee, M. C., Kendall, J. M., Bahrami, P., and Wang, T. G., "Sensational Spherical Shells," Aerospace America, 1986.

Bahrami, P. A. and Wang, T. G., "Analysis of Gravity and Conduction Driven Melting in a Sphere," J. Heat Transfer, 1986 (submitted).

Kendall, J. M., "Experiments on Annular Liquid Jet Instability and on the Formation of Liquid Shells," Phys. Fluids, 1986 (in press).

## Presentations

Lee, M. C., Schilling, C. H., Ladner, G. O., Wang, T. G., Bieg, K., and Wengert, P., "Direct Generation of Micro-balloons from Glass Melts for Inertial Confinement Fusion Target Application," presented at ICF Target Fabrication Specialist Meeting, Orlando, FL, March 1985.

## Influence of Convection on Microstructure

Clarkson University  
Dr. William R. Wilcox  
NAG8-480 (NASA Contact: F. Reeves, MSFC)  
June 1984 - June 1987

The objectives of this research are to determine the influence of convection on the microstructure of eutectics, to determine the longitudinal microstructure of directionally solidified Mn-Bi eutectic, and to measure transport processes in spin-up/spin-down of a Bridgman-Stockbarger ampoule.

Computer computations of the effect of convection on lamellar microstructure were extended to smaller volume fractions. The influence becomes smaller as the volume fraction deviates from 50%. An expression was derived for the influence of convection on the interfacial undercooling. New software is being developed for fibrous microstructures.

Experiments were completed on the influence of spin-up/spin-down on the microstructure of Mn-Bi eutectic. At small freezing rates the Mn-Bi phase forms irregular blades, which become larger and farther apart as the convection is increased. Furthermore the average composition of the solid deviates from the eutectic, increasing at the periphery and decreasing at the center until at low freezing rates no MnBi is found there. At intermediate freezing rates a fibrous microstructure occurs without stirring. As stirring is increased the fibers coarsen and eventually degenerate into irregular blades. As the freezing rate increases, the influence of convection on the microstructure diminishes.

The experimental results on Mn-Bi show a slightly larger effect for convection than predicted by the theory for lamellar eutectics. The theory predicts negligible effect for the mild convection produced by gravity, in contradiction to the experiments of Grumman Aerospace Corporation in which a two-fold change was observed by solidifying the material in space.

Experiments at intermediate freezing rates produced no change in the lead-tin lamellar spacing due to convection, in agreement with our theoretical prediction. However the rotation of the microstructure, presumably due to faulting, increased as the spin-up/spin-down rate was increased. Furthermore convection caused primary lead phase to form at the front of the ingot and primary tin at the end.

Techniques are being developed for pulling apart Mn-Bi ingots at elevated temperature, hopefully pulling the fibers out of the matrix. Some success has been obtained, but the

results are sensitive to temperature and strain rate. Etching techniques have also been investigated for revealing the longitudinal microstructure.

Spin-up/spin-down Bridgman-Stockbarger experiments were performed with an organic material. No fluctuations in interface position were observed as the rotation was started and stopped, indicating negligible perturbation of the heat transfer. Fewer gas bubbles were trapped in the solid when spin-up/spin-down was employed. An electrochemical apparatus is being constructed to permit mass transfer rates to be measured vs. time and radial position.

#### Publications

Lal, R. B., Aggarwal, M. D., Kroes, R. L., and Wilcox, W. R., "A New Technique of Solution Crystal Growth," Phys. Stat. Sol. 80, 547-552 (1983).

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Eisa, G. F., "Effect of Convection on the Microstructure of MnBi/Bi Eutectic Solidified from the Melt," J. Cryst. Growth, 1985 (in press).

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Popov, D. and Wilcox, W. R., "Influence of Convection on Spiral Structures of Lead-Tin Eutectic," J. Cryst. Growth, 1985 (in press).



## Modelling Directional Solidification

Clarkson University  
Dr. William R. Wilcox  
NAG8-541 (NASA Contact: I.C. Yates, MSFC)  
September 1985 - August 1988

The objective of this project is to elucidate phenomena of importance to directional solidification in space.

A unique heater was developed to permit the temperature profile to be varied in a vertical Bridgman-Stockbarger configuration. By increasing the temperature with height, convection could be almost entirely avoided. Use of an auxiliary heater near the solid-liquid interface caused convection to occur, with the degree of convection increasing as the temperature of the auxiliary heater was increased. Non-axisymmetric heating or tilting of the ampoule caused the convection cell to become non-axisymmetric. Variation of thermal conditions caused the flow to meander even though it was gentle.

Twinning was observed under the microscope during directional solidification of dodecanoic acid. The number of twins increased as the freezing rate and the temperature gradient were increased. Zone refining almost entirely eliminated twinning. Twinning was initiated by adding impurities whose molecules were similar in size and shape to the host. Foreign particles did not influence twinning.

Experiments are being planned on the influence of operating conditions, especially convection, in InSb-GSb alloys.

Behavior of a non-wetting liquid in partially filled ampoules was studied in the KC135 and via theory. In cylindrical ampoules the liquid separated into two or more columns, either separated by air or connected by a bridge of liquid along one wall. The bridge configuration was equivalent to a bubble on the opposite wall of the ampoule. Theory and experiment showed that the bubble was stable only up to a critical volume. In ampoules with triangular cross sections the liquid pulled away from the vertices and contacted the ampoule only along the centers of the three faces. Theory predicts that the contact width should decrease with increasing contact angle reaching line contacts at a critical contact angle.

## Publications

Rajendran, S. and Wilcox, W. R., "Steady State Thermal Modelling of Casting of Silicon," J. Cryst. Growth 69, 62-72 (1984).

Potts, H., and Wilcox, W. R., "Thermal Fields in the Bridgman-Stockbarger Technique," J. Cryst. Growth 73, 350-358 (1984).

Petrov, V., Rajendran, S., and Wilcox, W. R., "Steady State Thermal Modelling of Crystal Growth by the Bridgman-Stockbarger Method," Bull. Inst. Non-Ferrous Met. 20, 5 (1985).

Sen, R. and Wilcox, W. R., "Behavior of a Non-Wetting Melt in Free Fall: Experimental," J. Cryst. Growth 74, 591-596 (1986).

Rajendran, S., Wilcox, W. R., and Ravishankar, P. S., "Solidification Behavior in Casting of Silicon," J. Cryst. Growth 75, 353-366 (1986).

Potts, H. and Wilcox, W. R., "Chaotic Asymmetric Convection in the Bridgman-Stockbarger Technique," J. Cryst. Growth 74, 443-445 (1986).

Sen, R. and Wilcox, W. R., "Twinning of Dodecanedicarboxylic Acid," J. Cryst. Growth 75, 323-328 (1986).

Kingsley, J. P. and Wilcox, W. R., "Adsorption of Oxalate on Gibbsite," Hydromet. 16, 105-107 (1986).

Sen, R. and Wilcox, W. R., "Behavior of a Non-Wetting Melt in Free Fall: Theoretical," J. Cryst. Growth, 1985 (in press).

### 3. FLUID DYNAMICS AND TRANSPORT PHENOMENA

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Extension of Ostwald Ripening Theory

University of Alabama in Huntsville

Dr. James K. Baird

NAS8-35986 (NASA Contact: Dr. R. J. Naumann, MSFC)

October 1, 1984 - December 1, 1985

The objective of this investigation is to develop models based on the mean field approximation of Ostwald ripening to describe the growth of second phase droplets or crystallites. The models will include time variations in nucleation rate, control of saturation through addition of solute, precipitating agents, changes in temperature, and various surface kinetics effects.

Numerical integration schemes have been developed and tested against the asymptotic solution of Lifshitz, Slyozov and Wagner (LSW). A second attractor (in addition to the LSW distribution) has been found and, contrary to the LSW theory, the final distribution is dependent on the initial distribution. A series of microgravity experiments is being planned to test this and other results from this work.

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## Thermo-Diffuso Capillary Phenomena

Lewis Research Center  
Dr. A.T. Chai  
Dr. C.L. Lai  
Dr. R. Balasubramanian  
In-House

The objective of this program is to conduct fundamental microgravity research on interfacial surface tension driven motions by a temperature and/or concentration gradient.

The research being conducted involves two areas of interest: (1) thermocapillary convection and oscillation, and (2) motions of bubbles and droplets in a thermal and/or concentration gradient in a host fluid. In the former, progress has been made to simulate the temperature distribution along the surface of a thin layer of liquid sample when thermocapillary convection flow is taken into consideration. In the latter, work is being done to understand the effect of inertia on the thermocapillary motion of a spherical bubble. The parameters are such that the Reynolds number is large and the Marangoni number is small compared to unity. Under these conditions, there is a flow boundary layer near the surface of the bubble. The motion of the bubble is being analyzed in the presence of this boundary layer.

## Transport Processes in Solution Crystal Growth

Lewis Research Center  
Dr. A.T. Chai  
Dr. R. A. Wilkinson  
In-House

The objective of this effort is to conduct fundamental research in reduced gravity on transport processes occurring during solution crystal growth. Experimental techniques will be developed to monitor and control key parameters at the interface between a growing crystal and the solution from which it grows. Techniques developed in this investigation will lead to an in-space experiment on the shuttle. The focus of this research effort will be on non-incursive ground-based laboratory measurements of model systems and the definition of requirements for space experimentation.

Initially aqueous solutions will be used for easier control and instrumentation. Model systems with imposed steady flows and simplified boundary conditions will be studied for characterization. Various nonincursive measurement techniques are being investigated for proper applications in a newly built laboratory capable of low visualization, laser doppler velocimetry, Schlieren photography, specklegram, holographic interferometry, and Raman spectroscopy.

## Convection and Morphological Stability during Directional Solidification

National Bureau of Standards

Dr. S. R. Coriell

Dr. J. R. Manning

Dr. G. B. McFadden

Dr. R. J. Schaefer

H-27954B (NASA Contact: Roger Crouch, NASA HQ)

April 1985 - November 1986

The general aim of this task is the theoretical and experimental study of the fluid flow, solute segregation, and interface morphology which occur during directional solidification, including effects of gravity and microgravity. Space flight experiments, designed to determine cellular wavelengths as a function of growth conditions, are planned in collaboration with J. J. Favier and D. Camel of the Centre d'Etudes Nucleaires de Grenoble utilizing the directional solidification furnace being developed by the MEPHISTO Project.

During solidification of a binary alloy at constant velocity, thermosolutal convection can occur. Numerical calculations in two dimensions of the solute, temperature, and flow fields are being carried out for a variety of conditions, including microgravity with time dependent gravitational fields. The time dependent flow fields previously found for solidification vertically upwards with constant gravitational acceleration are being studied in detail in order to determine the conditions for non-steady flow. The effect of confining walls on the convection and solute segregation will be further investigated.

Experimental techniques for interface demarcation by Peltier pulsing will be examined, with the objective of finding the most effective methods to reveal interface shape development during microgravity solidification of metal alloys. By analysis of the heat flow and solute redistribution in the region of the solid-liquid interface, the optimum conditions of current density, pulse duration, and alloy composition will be sought to produce a visible trace of the interface shape with a minimum direct disturbance of the solidification process.

In the absence of convection, cellular morphologies are calculated numerically in two and three dimensions for alloys being considered for microgravity experiments. The effect of low fields on the morphology of the crystal-melt interface is investigated theoretically. This research, in collaboration with R. F. Sekerka of Carnegie-Mellon University and M. E. Glicksman of Rensselaer Polytechnic

Institute, elucidates the coupling between hydrodynamic and morphological instabilities.

#### Publications

Coriell, S. R., McFadden, G. B., Boisvert, R. F., Glicksman, M. E., and Fang, Q. T., "Coupled Convective Instabilities at Crystal-Melt Interfaces," J. Cryst. Growth 66, 514 (1984).

McFadden, G. B., Coriell, S. R., Boisvert, R. F., and Glicksman, M. E., "Asymmetric Instabilities in Buoyancy-Driven Flow in a Tall Vertical Annulus," Phys. Fluids 27, 1359 (1984).

Voorhees, P. W., Coriell, S. R., McFadden, G. B., and Sekerka, R. F., "The Effect of Anisotropic Crystal-Melt Surface Tension on Grain Boundary Groove Morphology," J. Cryst. Growth 67, 425 (1984).

Coriell, S. R., McFadden, G. B., Boisvert, R. F., and Sekerka, R. F., "Effect of a Forced Flow on Coupled Convective and Morphological Instabilities during Unidirectional Solidification," J. Cryst. Growth 69, 15 (1984).

Fang, Q. T., Glicksman, M. E., Coriell, S. R., McFadden, G. B., and Boisvert, R. F., "Convective Influence on the Stability of a Cylindrical Solid-Liquid Interface," J. Fluid Mech. 151, 121 (1985).

Coriell, S. R., McFadden, G. B., and Sekerka, R. F., "Cellular Growth during Directional Solidification," Ann. Rev. Mat. Sci. 15, 119-145 (1985).

McFadden, G. B., Coriell, S. R., and Boisvert, R. F., "Double-Diffusive Convection with Sidewalls," Phys. Fluids 28, 2716 (1985).

Glicksman, M. E., Coriell, S. R., and McFadden, G. B., "Interaction of Flows with the Crystal-Melt Interface," Ann. Rev. Fluid Mech. 18, 307-335 (1986).

McFadden, G. B. and Coriell, S. R., "The Effect of Fluid Flow Due to the Crystal-Melt Density Change on the Growth of a Parabolic Isothermal Dendrite," J. Cryst. Growth 74, 507 (1986).



## Theory of Solidification

Northwestern University  
Professor Stephen H. Davis  
NAG3-747 (NASA Contact: Dr. H. Gray, LeRC)

The research concerns the effort to understand on a quantitative level how various factors affect the morphology of a solidification front of a binary alloy. These factors include buoyancy-driven convection with and without Soret diffusion, phase-change convection, crystal and kinetic anisotropies and effects of bounding surfaces. The morphologies are directly connected to the solute distributions and hence the computations deliver the segregation fields as well.

The central theme is the understanding of the phenomena through the study of the instability behavior of the appropriate coupled systems. The coupling is among the interface, the melt and the solid and between the mechanisms of instability. The latter coupling modifies the individual instabilities and may produce new ones. The addition of new physical factors aims at generalizing the models toward more realistic systems.

## Suppression of Marangoni Convection in Float Zones

George Washington University  
Dr. Robert F. Dressler  
NAG1-325 (NASA Contact: A. Fripp, LaRC)  
January 19, 1983 - January 18, 1986

The objective of this research is to demonstrate, by means of a 1-g experiment, that the idea of space processing to use tangential gas jets for suppressing the unwanted thermal-capillary (Marangoni) convection always present in a float zone, is valid and efficacious.

For proposed processing of highly reactive semiconductor materials, e.g. silicon, in microgravity ( $g$ ), although the thermal-buoyant convection will be suppressed in  $g$ , this will not reduce the Marangoni convection since there must always be a temperature gradient, hence a surfacetension gradient, in a float zone. Our idea for space processing is to blow jets of a non-contaminating gas, e.g. argon or xenon, tangentially over its free molten surface to establish a shear stress to counterbalance the surface-tension shear which excites the Marangoni convection. Since the principle involved is identical, our earth-based experiment uses an air jet and a transparent silicone oil in a half-float zone configuration to demonstrate that the Marangoni convection can be significantly reduced by our method. There are three major difficulties due to 1-g in our experiment, but in spite of these, we have attained an average reduction of 66 to 75% in the Marangoni velocities, showing our idea is workable. We are now engaged in the initial planning for a new project, using a middeck Shuttle experiment, in which all three 1-g problems will be eliminated. Therefore, we expect our anticipated  $g$  experiment will attain reductions better than about 98%. This will then indicate use of our method for commercial fabrication of semiconductors in the Space Station.

## Transient Heat Transfer Studies in Zero G

National Bureau of Standards - Boulder Laboratories

Dr. Patricia J. Giarratano

Dr. Vincent D. Arp

H-27954

October 1984 - November 1985

The objective of this work is to provide computer codes and/or correlations for application in the design of transient heat transfer systems near thermodynamic critical conditions (highly compressible fluids). The model includes the effect of motion induced by the thermal expansion of the fluid adjacent to the flat geometry heater surface, and predicts the temperature profile in the fluid during a heat pulse. The low gravity environment of space provides the opportunity to study the thermally induced motion and other near critical heat transfer mechanisms which may be masked by gravity driven convective motion on earth. Experimental data have been acquired both in the laboratory and during two flights on the NASA KC-135. Mach-Zehnder interferometry was the measuring technique used to study the temperature fields in the heated fluid. In the course of the experimental work the inherent limitation of Mach-Zehnder interferometry for quantitative evaluation to steep gradient conditions, such as exist in the thermal boundary layer during the transient heat transfer to the highly compressible near critical fluid, became an obstacle to measurements over a wide range of conditions. A summary of this work has been written and is currently in NBS editorial review.

In light of the above noted limitations of Mach-Zehnder interferometry for temperature and/or concentration measurements within the boundary layer, we are exploring the applicability of holography for these measurements. These exploratory measurements in our laboratory (and if demonstrated to be applicable) on the KC 135 will be conducted in association with MSFC.

### Publications

Giarratano, P. J., Arp, V. D., and Kumakawa, A., "Limits to Optical Measurements of Heat Transfer in High Gradient Systems," in press.

Center for Microgravity Fluid Mechanics and Transport Phenomena

University of Colorado  
Dr. D.R. Kossy  
Dr. R. Sani

The purpose of the Center will be to develop a unique expertise for modeling and experimental analysis of fluid systems located in a low-gravity environment. Research programs carried out in the Center will be of an interdisciplinary nature, involving expertise from several different engineering areas, from solid state physics and from mathematics. An outreach program will be used to communicate research advancements to industry, government and academic institutions and to provide input to analyze low-gravity fluid systems. It is anticipated that the Center will become a national resource for industries and government laboratories concerned with space-related fluid processes.

Frequent and reliable access to the near-earth space environment via the shuttle, and the forthcoming space station, provides the scientific and technological communities with a unique opportunity to investigate novel physical phenomena. The low levels of both gravitational acceleration and ambient pressure permit one to manipulate system processes in ways not possible in ground-based facilities. As a result there is a growing industrial awareness that the microgravity environment of space can be used for developing new materials, products and technologies. At the same time the scientific community has recognized the opportunity to reassess the effect of gravity on diverse phenomena, examples of which include crystal growth, combustion of gases, liquids and solids, formation of polymers, glasses and alloys, solidification of melts, and the spreading of liquid films on solids.

A Study of Flow Boiling in Micro and Zero Gravity Environments

University of Houston  
Professor John H. Lienhard  
Professor Roger Eichhorn  
NAG3-537 (NASA Contact: R.M. Vernon, LeRC)  
June 16, 1984 - July 31, 1986

The objective of this research is to establish criteria for identifying the regions in which gravity does, and does not, influence the peak flow boiling burnout heat flux. A secondary objective is to begin work on identifying the way in which gravity influences burnout, when its influence is present.

We have at our disposal two experimental tools for identifying the ranges of relevant parameters (liquid flow velocities, heater sizes, thermal properties of the liquid, etc.) in which gravity does and does not influence burnout. One of these is a flow loop in which the liquid may be run in either an upward or downward direction over a heating element. The other is a large centrifuge apparatus in which we have installed a complete flow loop. We also have at our disposal the tactic of attempting to develop a firm analytical understanding of the burnout process. A correct rational prediction of burnout will include (or not include) the influence of gravity and display what its effect upon the process will be.

Our work to date has involved the use of the up-down flow loop to discriminate the role of gravity and to provide accurate diagnoses of several aspects of the boiling process upon which predictions may be built. These include matters such as vapor removal configuration, the character of the Helmholtz instability process that leads to instability, the relation of the burnout process to alternating current forcing, when it is used, and so forth.

The work has also led to the design and construction of a miniaturized (2 m dia.) flow loop to ride in the centrifuge. This apparatus is being used to establish the gravity scaling of the boundary between the regions of gravity influence and gravity noninfluence. Finally, the analytical work has led to the best understanding, to date, of the mechanism of burnout during flow boiling across horizontal cylinders.

## Publications

Kheyrandish, K., Dalton, C., and Lienhard, J. H., "A Model for Fluid Flow During Saturated Boiling on a Horizontal Cylinder," J. Heat Transfer, 1986 (in press).

Kheyrandish, K. and Lienhard, J. H., "Mechanisms of Burnout in Saturated and Subcooled Flow Boiling over a Horizontal Cylinder," J. Heat Transfer, 1986 (submitted).

The Effects of Microgravity on Laser-Induced Gas-Phase  
Aerosol Polymerization

NASA Langley Research Center  
Dr. Willard E. Meador  
Gilda A. Miner  
In-House  
April 1, 1986 - April 1, 1987

The objectives of this research are: (1) to enhance understanding of basic polymer science by investigating polymerizations performed without solvents (aerosol phase), thereby eliminating solvent effects and permitting a greater and more detailed understanding of basic mechanisms, and (2) to determine the effects of microgravity on the composition, structure, and mean molecular weight of photoinitiated polymers.

The primary research is to establish a gas-phase/aerosol laser polymerization data base in a laboratory environment. Polymerization will be accomplished and measurements made of the rate of polymerization, yield vs. time, mean molecular weight, and dependences on mixing ratio and laser parameters. These data will be used in developing a model for predicting characteristics of the same polymers to be formed in the microgravity environment of space.

A Fundamental Study of Nucleate Pool Boiling Under  
Microgravity

University of Michigan  
Dr. Herman Merte, Jr.  
NAG3-663 (NASA Contact: R.W. Vernon, LeRC)  
October 5, 1985 - November 30, 1986

The objective of this research is to determine by experiment, and develop analytical models to predict heat transfer and vapor bubble dynamics associated with nucleation, bubble growth/collapse and motion for nucleate pool boiling in microgravity.

The study proposed seeks to improve the understanding of basic processes that constitute boiling in the absence of buoyancy effects, which mask other phenomenon, and is a component in the development of a data base for space applications. This is accomplished by a combination of theoretical analyses, laboratory testing at standard gravity, plus testing in a ground-based reduced gravity environment. These will provide a basis for defining the requirements for flight testing in orbit.



Experimental and Theoretical Studies of Wetting and Multilayer Adsorption

National Bureau of Standards

Dr. M. R. Moldover

Dr. J. W. Schmidt

Dr. J. W. Cahn

Dr. R. F. Kayser

H-27954B

April 1977 - continuing task

Our recent studies of a binary liquid mixture (isopropanol-perfluoromethylcyclohexane) has established that a first order wetting transition exists at the vapor-liquid interface. However, the theoretically predicted prewetting line associated with this transition has not been detected even though our experiments on this system have been the most carefully controlled to date. In another system (methanol-cyclohexane), we have shown that the vapor liquid interface can be stabilized with the addition of deuterated cyclohexane. Near the critical point strong stable ellipsometric signals were obtained both above and below  $T_c$ .

On other work, films of  $SF_6$  in fused silica have been investigated in a unique experiment in which we find good agreement with the theory of dispersion forces when retardation effects and gravity are taken into account. There are no parameters to be adjusted upon comparing the experiments and the theory.

A theory has been worked out for the effect of surface ionization on wetting layers. A theory has been worked out for the effect of capillary waves on surface tension.

Publications

Kayser, R. F., Schmidt, J. W. and Moldover, M. R., "Wetting Layers and Dispersion Forces for a Solid in Contact with a Vertical Wall," Phys. Rev. Lett. 54, 707 (1985).

Schmidt, J. W. and Moldover, M. R., "The Liquid Vapor Interface of a Binary Liquid Mixture near the Consolute Point," J. Chem. Phys. 83, 1829 (1985).

Moldover, M. R. and Schmidt, J. W., "Wetting, Multilayer Adsorption and Interface Phase Transition," Physica 12D, 351 (1984).

Kayser, R. F., Moldover, M. R., and Schmidt, J. W., "What Controls the Thickness of Wetting Layers?" J. Chem. Soc. Faraday Trans. II, 1985 (accepted).

Kayser, R. F., "The Effect of Capillary Waves on Surface Tension," Phys. Rev. A, 1985 (accepted).

Kayser, R. F., "The Effect of Surface Ionization on Wetting Layers," Phys. Rev. Lett., 1985 (submitted).

Energy Stability of Thermocapillary Convection in Models of the Float-Zone Process

Arizona State University  
Professor G. Paul Neitzel  
Professor Daniel F. Jankowski  
NAG3-568 (NASA Contact: Dr. A.T. Chai, LeRC)  
August 5, 1984 - January 3, 1987

The objective of this research is to apply energy-stability theory to models of the float-zone, crystal growth process, thereby obtaining lower bounds on the critical Marangoni numbers for the onset of oscillatory convection which degrades crystal quality.

The float zone, crystal growth process exhibits a basic state of thermocapillary convection due to temperature (and hence, surface tension) gradients along the free surface of the melt. Energy stability theory is a means of obtaining a stability bound for this basic state, i.e., below the energy limit (in this case a value of the Marangoni number) any disturbances to the basic state must decay. This information is useful since it identifies regions in the appropriate parameter space where undesirable oscillatory convection cannot occur. The calculation of the energy limit is a two-step process: (1) numerical determination of the basic state, and (2) nonlinear computations to obtain the stability limit. Step 1 has been completed for a model half-zone by means of both finite difference and finite element methods. Currently work is proceeding on the corresponding second step.

Publications

Squire, T. H., Shen, Y. H., Neitzel, G. P., and Jankowski, T. H., "Thermocapillary Convection in a Model Float-Zone," Phys. Rev., 1985 (accepted).

## Thermocapillary and Diffusocapillary Migration of a Fluid Drop

University of Colorado  
Professor Robert L. Sani  
NAG3-493 (NASA Contact: A.T. Chai, LeRC)  
November 1, 1983 - October 30, 1986

The migration of bubbles, or drops, plays an important role in many engineering science and space manufacturing problems. In material science processes as in the manufacturing of glasses, etc., gas bubbles can be formed from the by-products of chemical reactions or gas trapped in the interstices of the raw material. In the low-g environment of space, forces other than gravitational must be utilized as a bubble separation technique. It is well-known that gradients in interfacial tension on the bubbles' surface can promote droplet motion in the direction of decreasing interfacial tension and hence provide such a separation mechanism. Thus, the role of thermocapillary and diffusocapillary migration of a bubble, or drop, can be of paramount interest in materials processing in space.

Ongoing research is focused on the quantitative investigation of such processes by analytical and numerical models. The initial phase of the study dealt with a thorough literature search for pertinent studies on thermocapillary and buoyancy driven bubble migration and an assessment of this literature. Computer algorithms for the available analytical solutions of interest (primarily the thermocapillary migration of a spherical bubble [or bubbles] in Stokes flow) have been developed so that effects of various physical parameters can be easily studied. Such existing analytical solutions are being extended to model concentration effects and in particular the effect of surface active material and to account for some convective transport effects. Concurrently, the development of a Galerkin-finite element algorithm for the discretization and numerical solution of axisymmetric bubble migration problems has been initiated so that significant nonlinear convective transport and surface deformation effects can be investigated. Initially, combined buoyancy and surface tension driven flows in planar and cylindrical geometries are being investigated as benchmark cases while appropriate general mesh adaption techniques to track the free surface are developed.

## Publications

Engelman, M. S. and Sani, R. L., "Finite Element Simulation of Temperature Dependent Free Surface Flows," in Proceedings of Fourth International Symposium on Numerical Methods in Thermal Problems, Swansea, UK, 1985 (in press).

The Roles of Fluid Motion and Other Transport Phenomena in  
the Morphology of Materials

Princeton University  
Professor Dudley A. Saville  
NAG3-447 (NASA Contact: Dr. A.T. Chai, LeRC)  
July 15, 1983 - July 12, 1986

The purpose of this investigation is to develop, and later test, models describing the ways transport phenomena influence the morphology of crystalline materials. Specific emphasis will be placed on processes involving phase change due to the growth of dendritic forms.

Two tasks are under way, one is a study of the effects of convection on morphological stability. The first part of this work involves extension of the theory developed by J. S. Langer for low undercoolings (low Peclet numbers) to encompass large undercoolings. With this as a reference state, effects of fluid motion will be investigated theoretically.

The second aspect of the work is a study of the effects of convection on different techniques for growing protein crystals. This work is a combination of theoretical and experimental work using various model compounds. Current experimental work is focused on interferometric studies of lysozyme growth.

The Study of Electromagnetically Driven Flows in Molten Salts Using a Laser in a Microgravity Environment

Massachusetts Institute of Technology  
Professor Julian Szekely  
David Forrest  
NAS3-24642 (NASA Contact: Fred Harf, LeRC)

The objective of this research is to develop the feasibility of studying electromagnetically driven flow in molten salts, using laser velocimetry, in a microgravity environment. The main motivation for doing this in microgravity is due to the fact that under earthbound conditions the joule heating in the poorly conducting salts would give rise to buoyancy driven flows, which would become dominant.

The work was initiated recently. The initial work consisted of performing scoping calculations in order to design the experimental apparatus, such that reasonable, measurable flows will be produced but at the same time excessive overheating is avoided. Preliminary work has also been carried out to identify suitable electrodes, container materials and salt systems.

## **4. BIOTECHNOLOGY**



## Biomedical Products from Culture Media in Space

Baylor College of Medicine  
Professor M. Zouhair Atassi  
NAS9-17403, Tasks 5 and 6 (NASA Contact: D.R. Morrison, JSC)  
June 1, 1985 - May 31, 1988

The objectives of the first task are to: (1) analyze culture media from space flight experiments for their amino acid compositions, in order to optimize the composition of these media for better support of cell growth and proliferation in future zero gravity experiments, and (2) synthesize selected regions of urokinase.

Continuous flow electrophoresis in microgravity has been employed in the separation of kidney cells into subgroups. These subgroups appear to produce various amounts of urokinase. The assay for urokinase now depends on measurement of enzymic activity. This is not a very sensitive method and it would also be completely unable to detect and measure the inactive form of the enzyme (i.e., the pro-enzyme), which is the form initially produced and then undergoes alteration to yield the active enzyme. In order to better monitor kidney cell separation and obtain a more precise correlation with viability and activity of the various cell groups, a very precise method for measurement of urokinase is needed. Also, it is necessary to measure the amounts of both the pro-enzyme as well as the active enzyme. The purpose of this task is to develop radioimmunoassays (RIA) for measurement of both forms of the enzyme. Polyclonal and monoclonal antibodies will be made with specificities directed against preselected regions of urokinase. These preselected regions are made synthetically and the synthetic peptides are used as immunogens to obtain antibodies of the desired specificities. These antibodies will be used to develop RIA and ELISA assays for urokinase and pro-urokinase.

Using high pressure liquid chromatography (HPLC) and amino acid analysis, 154 samples of culture media have been analyzed. The results have been delivered to NASA(JSC) and a computer analysis of the data is now being carried out by NASA scientists. Ten peptides, representing various regions of urokinase and pro-urokinase, have been synthesized. Two peptides have already been injected into mice and antibodies that bind urokinase have been obtained. The other eight peptides are in various degrees of completion of synthesis or purification.

Activities to be completed are: (1) analysis pre-flight, in-flight, and post-flight culture media supplied by JSC; (2) synthesis and/or purification of urokinase

peptides; and (3) preparation of mouse and/or rabbit antibodies against urokinase peptides.

The objective of the second task is to develop RIA, ELISA and fluorescent methods for quantitative measurements of IR on cells.

Separation of pancreatic alpha and beta cells have been obtained by the CFES method in zero gravity. However, it is not possible to determine on the alpha cells the effect, if any, of the methods of separation and manipulation on the insulin receptor (IR) content. Therefore, a very sensitive assay is needed that will enable the monitoring of IR density on the alpha cells during the various stages of cell separation, manipulation and culture. Clearly, the most sensitive approach would be an immunochemical method that will employ specific anti-IR monoclonal or polyclonal antibodies. However, since IR is a ubiquitous membrane protein occurring in animal cells of various tissues, but in differing densities, the anti-IR monoclonal antibodies can be employed as the basis for a new method of separation of various cells using CFES. Cells tagged with anti-IR antibodies will be expected to migrate in different mobilities in an electric field, depending on the IR density on the cell membrane. In addition, an RIA for the IR will have an important diagnostic application in detection of type II (insulin-resistant) diabetes, on the basis of low levels of insulin receptor on circulating blood monocytes and lymphocytes.

By computer analysis of the covalent structure of human IR, seven regions of the molecule were selected as ideally suited to be synthesized and used as immunogens for the preparation of specific anti-IR monoclonal antibodies. These peptides have been synthesized and are being purified.

Activities to be completed are: (1) synthesis and purification of human IR peptides; (2) preparation of mouse antibodies against the IR peptides; and (3) testing the IR peptides for their binding activity to insulin to human autoantibodies against IR.

## Research in Biological Separations and Cell Culture

University of Texas Health Science Center at Houston

Dr. Grant H. Barlow, BRCH

Dr. Baldwin H. Tom, BRCH

Dr. Lynne P. Rutsky, U.T. Medical School

Dr. L. Scott Rodkey, U.T. Medical School

NAS9-17403 (NASA Contact: D.R. Morrison, JSC)

June 1985 - September 1988

The objectives for the research at the Bioprocessing Research Center at Houston (BRCH) center around development of bioseparation science and technology in partial support of NASA's efforts in bioprocessing. Within this context, the research focuses on four interrelated objectives:

(1) support and utilization of the McDonnell Douglas continuous flow electrophoresis system (CFES); (2) development of cell candidates for electrophoresis separation; (3) development of recycling isoelectric focusing for cell products; and (4) development of biochemical analysis of cultured cell products.

To accomplish the first objective, a laboratory area has been developed to support continuous flow electrophoresis studies, using not only the CFES from McDonnell Douglas, but other instrumentation which complements the CFES. Included in the bioseparations studies will be comparative studies using HPLC instrumentation as well as other free flow electrophoresis systems. The most effective material separation conditions will be determined in ground based studies that might be translated into use on the shuttle-based CFES. Target materials for separation include soluble hormones and proteins, and whole cells and subcellular particles.

To accomplish objective 2, a series of human cell model systems have been chosen to develop the optimal conditions for their separation. The model cell systems include the human embryonic kidney cell, with its bioproducts of erythropoietin, urokinase and tissue plasminogen activator; colon cancer cells will serve as a model for the production of tumor effector materials, including carcinoembryonic antigen (CEA), immunoregulatory factors and immunogenic cell membrane proteins; and finally hybridoma cells will be evaluated for their separation by electrophoresis. They will be monitored by the separated cell's ability to produce monoclonal antibody. The rationale for studying the monoclonal, hybridoma cell, which on first approximation would seem to be a fruitless project, is based on recent observations that there are chromosomal rearrangements and losses in clonal cell populations which lead to changes in the nature of the monoclonal antibody produced. Furthermore, if an efficient separation technique could be

utilized, a potentially faster screening may be found for the cloning of hybridoma cell populations.

To accomplish objective 3, a recycling isoelectric focusing unit has been purchased. This unit was designed and is patented by Milan Bier of the Separation Sciences Center at the University of Arizona in Tucson. Additionally, narrow range ampholytes necessary in the isoelectric focusing studies have been prepared for evaluation in separating closely related macromolecules. All separation techniques will be compared to maximize efficiency of purifying cells and cell products under varying conditions.

To accomplish objective 4, human embryonic kidney cells will be used as a source of plasminogen activator, urokinase, erythropoietin, and possibly human granulocyte stimulating factors for purification and assay development in support of the microgravity research on the NASA contract. Purity of cell fractions will be determined both by their functional capabilities in secreting specific materials, and also by the use of available monoclonal antibodies identifying specific cell types.

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## Center for Separation Science

University of Arizona

Dr. Milan Bier

NAGW-693

January 1, 1985 - December 31, 1988

The Center for Separation Science has been organized to promote industry awareness and involvement in the NASA program on biotechnology in space. Up till now the Center has mainly built on the pre-existing expertise in electrophoresis. Through generous contribution of instrument manufacturers and at no cost to NASA, the Center has built a unique facility for preparative and analytical electrophoresis.

The instrumentation, some made in the Center's own laboratory, other manufactured in Sweden, Germany, England, and Michigan, includes all the best known commercially available electrophoretic instruments. Emphasis is on preparative applications, usable on a full range of scale, from microgram to kilogram. Most of these instruments were never before available in one laboratory. This instrumentation is backed by professional expertise in all major areas of electrophoresis, encompassing two-dimensional analytical electrophoresis, isotachopheresis, isoelectric focusing, and computer modeling.

The Center is attracting a great deal of interest from the pharmaceutical and genetic engineering industries and is propagating its technology through publications, organization of workshops and conferences, and visits from individual researchers.

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## Protein Crystal Growth in Low Gravity

Stanford University, Center for Materials Research  
Professor Robert S. Feigelson  
NAG8-489 (NASA Contact: V. Yost, MSFC)  
August 9, 1984 - August 8, 1987

The structure of proteins is of great interest to biological scientists. A number of diffraction techniques are available to determine these structures, but they require single crystals of the protein of interest. Obtaining these crystals with suitable size and perfection has proved to be a bottleneck. There is a reasonable expectation that growth in a low gravity environment would improve the size and perfection of protein crystals. The ultimate goal of this project is to design such an experiment. However, it is also necessary to understand the mechanisms of protein crystal growth and what parameters influence growth in order to be able to design the growth experiment. The research in this project is designed to gain this understanding.

Canavalin has been selected as a model protein. It is being grown by changing the pH of the protein solution by direct addition of acid. Kinetics and growth are being studied microscopically at various pHs, protein concentration, and temperatures. Flow has an important influence on crystal growth and the concentration-induced flows around the growing canavalin crystals are being studied using Schlieren techniques. Research will also be done on the concentration of both the protein and hydrogen ion versus time during growth. The protein concentration gradients within the boundary layer of the growing crystal will be investigated, as will the prenucleation clusters found in the solution. All of this data will be combined to determine a model for the growth of protein crystals. This model will be used to optimize the growth conditions. The information gathered in the earth-based experiments will also be used to design a space flight experiment to gather the same type of data in a low gravity environment.



Bioreactor Development for Use in a Microgravity  
Bioprocessing Environment

Rice University

Dr. Larry V. McIntire

Dr. E. Terry Papoutsakis

Dr. Ka-Yiu San

NAS9-17403, Task 8 (NASA Contact: D.R. Morrison, JSC)

June 1, 1985 - May 31, 1988

The objective of this research is to study the hydrodynamic effects on cells and mixing as well as control aspects of a cell-culture bioreactor for space bioprocessing. The emphasis is almost exclusively on engineering aspects of the cell culture bioreactor. The objectives and results are described in two parts.

I. Fluid Mechanics and Mixing in Cell Culture  
Bioreactors

Most normal (non-malignant) tissue cells are anchorage-dependent, requiring a solid surface to adhere to in order to grow. One effective approach is to grow the cells on microcarriers, small polymer beads that may be suspended by agitation in tissue culture medium to provide an environment with high surface area per volume and nearly homeostatic conditions for the cells. However, the agitation required to prevent the cell-covered beads from settling out of the medium may also cause cell damage and death. The microgravity environment of space obviates the problem. However, a fundamental understanding of the effect of agitation on cells does not exist, making it difficult to design a space bioreactor that takes maximum advantage of microgravity.

Experimental work is now underway in two bioreactors to confirm some of the predictions. The emphasis is on the design and operation of the reactor system, rather than on the biology of how the cell responds. Some of the variables considered to be important in this work are the detailed agitator design (number of blades, diameter, pitch, etc.), agitator rotational speed, medium viscosity, and micro-carrier density, diameter and concentration.

Analysis of the ways in which cells on the surface of a microcarrier bead might be damaged by excessive agitation requires that three distinct hydrodynamic regions in the bioreactor be considered. These are the turbulent bulk liquid, the laminar boundary layers around solid objects in the reactor, and collisions with parts of the reactor or other beads. In typical systems the bulk liquid is turbulent because of the disturbed flow around the impeller and the various probes and sample lines that also act as baffles. In isotropic turbulence the kinetic energy of the

turbulence is passed from large eddies to smaller eddies without loss. In a laminar boundary layer the cells on the bead are relatively protected. The bead tends to rotate at about 20 Hz, and the net effect of the forces resulting from rotation in a shear field is to move the bead away from the solid surface. Collisions of beads against solid objects in the reactor occur when the beads' centers pass within one bead radius of the object. From a knowledge of the flow pattern around a rounded leading edge, it can be calculated that a typical bead hits the impeller approximately once every four minutes. Collisions with other solid objects should be less important because the lower fluid velocity past them reduces both the frequency and the kinetics energy of the collision. Compared to bead-bead collisions which occur to each bead every five seconds, the frequency of impeller collisions is 1/40 as great but the kinetic energy of the collision is 2500 times larger because of the greater relative velocity.

The effects of agitation on cells growing on micro-carriers are proposed to be caused by turbulence of the size of the beads and/or by collision of the beads against other objects. Literature data support the turbulence size hypothesis, but do not allow any conclusions to be made about collision effects. The observed changes over the course of the runs in the number of confluent beads, bare beads (those with no cells attached) and bridges between beads suggest that analysis of the data in terms of population dynamics would be fruitful. This could also explain why some cultures reach a maximum of, for instance,  $3 \times 10^5$  cells/ml but a fresh culture at the same conditions seeded at  $3 \times 10^5$  cells/ml would grow to higher densities. Over the next year we intend to investigate the effect of medium viscosity, agitator speed and agitator design on the growth of BEK cells. The data will be analyzed in terms of growth rate and population balances. The goals are to understand the specific mechanism(s) by which individual cell growth is inhibited, how that affects the total cell population, and whether the reduced net growth is caused by a lower intrinsic growth rate or by a death rate acting on an actively growing population.

## II. Control of Bioreactors

The objective of this work is to develop an expert system-based intelligent control scheme for the space bioreactor. The design and implementation of effective control strategies for bioreactors are often hindered by the lack of detailed knowledge about the system dynamics and the unavailability of proper sensors. In addition, the control of bioprocesses is often complicated by the possibility of inherent time dependent variations in process characteristics and parameters. It is thus the long range goal to develop a control scheme which is capable of combining

information from process model, operator's experience, and previous experimental data base for the full automation and effective control of bioreactors.

The research effort focuses on the fundamental studies of the control of bioreactors and the feasibility study, development, and implementation of an expert system-based bioreactor control scheme. Goals for the past year included: (1) studies on the fundamental aspects of the control of bioreactors with special emphasis on the observability and controllability criteria, and (2) feasibility studies on the proposed control scheme.

Preliminary results from the study on the fundamental aspects of the control of bioreactors indicate that such systems are at best weakly controllable and possibly not totally observable. More work in this direction is in progress. A study aiming at the investigation of the feasibility and the impact of the addition of an expert system to the conventional controller is in progress. For a fixed gain controller with possible system dynamic variation, the controller may be either too tightly or too loosely tuned which results either in controller instability or sluggish response. An expert system based controller is capable of predicting the reactor buffer content and thus is able to pick the correct controller setting. This results in a far better controller performance.

The research will continue to study the fundamental aspects of the control of bioreactors and to confirm the preliminary results of this study through rigorous theoretical analysis and a series of computer simulations. Results from this study will form the fundamental foundation for the proposed expert system-based control scheme.

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## Development of a Space Bioreactor

Johnson Space Center

Dr. Dennis R. Morrison

Dr. David Wolf

Dr. T. Papoutsakis, Rice University

Dr. K. Y. San, Rice University

Dr. R. Nerem, University of Houston

Dr. C. Goochee, University of Houston

In-Center

January 1983 - continuing task

The effort to develop a space bioreactor continues a long-term NASA commitment to biological research and development in space. The earliest efforts showed that some biological substances can be better purified in space using the continuous-flow electrophoresis system (CFES) devised by McDonnell Douglas. The CFES also separated living mammalian cells into fractions, some of which produced larger amounts of medically important substances than others. These results led to the concept of a space bioprocess. Such a process would start with a CFES separation of cells followed by the culturing of the cells secreting more of the desired drug. The drug could then be isolated from the culture medium and purified by another CFES separation. The ability to culture cells in space would have far reaching consequences. For example, one-time-only experiments have shown that plant cells are sensitive to weightlessness. A space bioreactor would allow scientists to conduct an ongoing series of experiments to understand this sensitivity. Such a bioreactor could be one component to a permanent biological research facility in the Space Station.

In the past year, a bioreactor has been built in the laboratories of the NASA Medical Sciences Division at the Johnson Space Center. The design of this reactor represents a significant improvement over bioreactors now in use. This improvement is partly to take advantage of the conditions of space mentioned previously and partly to satisfy criteria imposed by the design of the Space Shuttle Orbiter. Criteria that could result in spinoff technology are compactness, energy efficiency and automatic operation with minimum operator (astronaut) attention. The basic design is a vessel containing the cells connected to a loop in which a nutrient medium circulates. The loop leaves the vessel through a filter that keeps the cells in the vessel. It returns after oxygenation and nutrient replenishment. In a second loop, accessible from the first, is a high molecular weight filter systems designed to periodically recover concentrated culture medium containing the cell products. The space bioreactor lab test unit has recently been completed and tests with kidney cells attached to microcarrier beads have already begun. The objective for

next year is to test and improve the design and to definitize operating procedures for the first flight test.

The project goal is to have a space bioreactor ready for Space Shuttle flight in October 1988. This Space Shuttle middeck flight bioreactor will be designed to sustain human kidney cell culture. It will produce a solution of urokinase, an enzyme that participates in the dissolution of blood clots. This protein is representative of many drugs that could be produced by the same process. Culturing of mammalian cells is difficult because they are fragile. The culture of human cells in space is expected to give opportunities for exploration of potential benefits such as mixing with very low shear, unique oxygen delivery and better control of the microenvironment around the cells. Whenever possible, components of the ground bioreactor will be flight-certified and incorporated into the flight unit. The long-range program goal is to incorporate the Space Shuttle experience into the design of the Space Station bioreactor facility.

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## Fluid Dynamic Studies in Cell Bioreactors

University of Houston

Dr. Robert M. Nerem

Dr. Stanley Kleis

Dr. Murina J. Levesque

NAS9-17403 (NASA Contact: D.R. Morrison, JSC)

June 1985 - May 1988

The objective of this research includes determining the detailed fluid dynamic characteristics of mammalian cell bioreactors and the influence of hydrodynamic forces on the structure and function of mammalian cells such as employed in bioprocessing. The research are focused by these activities being specifically related to the space bioreactor which the NASA Johnson Space Center plans to fly late in this decade.

The first objective of studying the detailed fluid dynamic characteristics of a mammalian cell bioreactor has emphasized specifically stirred tank reactors. There are several different versions which are being considered by NASA for their flight program. The studies will include both flow visualization measurements to determine the general features of the flow field as well as Laser Doppler Velocimetry (LDV) to measure point velocity in localized regions. The former measurements are being developed in such a way to include the application of the flow visualization technique to measurements during space flight. The latter point-velocity LDV measurements will be employed to determine the specific characteristics of the flow field including the shear stress to which cells will be exposed.

The influence of hydrodynamic forces on mammalian cells will be studied in the cell culture laboratory of the Bioengineering Research Center. For shear stress studies, cultured monolayers of cells will be exposed to known levels of shear stress, both steady and pulsatile, using a parallel plate flow chamber. Although to date only endothelial cells have been employed, other cell types including human embryonic kidney cells will be studied in the future. Effects of pressure on cell structure and function will also be studied in the cell culture laboratory.

## Containerless Polymeric Microsphere Production/Biotechnology

Jet Propulsion Laboratory  
Dr. W-K. Rhim  
Dr. Michael T. Hyson  
Dr. Manchum Chang  
NAS7-918  
October 1985 - September 1987

The objective of this task is to investigate containerless production of monodisperse polymeric microspheres for biomedical applications. Microspheres of precise sizes and specific properties are needed for medical diagnostic tests, chromatography, cell sorting, and cell labelling. The uses and economies involved in the mass production of biomedically valuable microspheres in space will be investigated.

Using a ground-based electrostatic levitator, various monomer or polymer droplets will be injected, levitated, polymerized, and collected. Conditions needed for polymerization, monodispersity, the physical and chemical properties and their utilities for various biomedical purposes will be determined. Information derived from these experiments will be used to assess the feasibility of mass producing microspheres in the space microgravity environment. In addition, using monodisperse HEMA particles we produced, antibody binding tests will be carried out followed by affinity separations of bone marrow cells, separations of natural killer lymphocytes, and cell labeling by fluorescent particles.

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## Research and Technology for Isoelectric Focusing

University of Texas Health Sciences Center

Dr. L. Scott Rodkey

NAS9-17403 (NASA Contact: D.R. Morrison, JSC)

June 1, 1985 - May 31, 1988

The objective of this work is to provide background information for Johnson Space Center relating to rapid and efficient separation of proteins by recycling isoelectric focusing. Subprojects within this overall objective are to (1) evaluate the recycling isoelectric focusing instrument available from Ionics, Inc. for its capacity to separate proteins, specifically antibodies, into discrete homogeneous subpopulations and (2) to develop methodologies for synthesis of inexpensive narrow-range ampholytes for use in isoelectric focusing.

A recycling isoelectric focusing instrument has been purchased from Ionics, Inc. and has been installed in the laboratory. Optimal running conditions will be determined by varying the several parameters that govern the speed of separation and resolution of the instrument. The second objective is being approached by chemical modification of available polyethylenepolyamines in order to create structures which will provide suitable ampholyte mixtures that will cover selected narrow ranges when subjected to electrical fields. The RIEF and the narrow range ampholytes will be used to fractionate polyclonal antisera for production of monoclonal antibodies.

### Publications

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Knisley, K. and Rodkey, L. S., "Affinity Immunoblotting: High Resolution Isoelectric Focusing Analysis of Antibody Clonotype Distribution," J. Immuno. Methods, 1986 (submitted).

## Cell Concentration Limits in Electrophoresis

University of Texas Health Science Center  
Dr. Lynne Rutsky  
NAS9-17403 (NASA Contact: D.R. Morrison, JSC)  
June 1985 - June 1987

In previous studies using the Continuous Flow Electrophoresis System (CFES), it has been observed that injecting too high a concentration of living cells will reduce the bandsread and compromise the resolution of the separation. For example, the bandsread (i.e., effective electrophoretic mobility distributions) of human kidney cells are reproducible up to sample concentration of  $2 \times 10^7$  cells/ml. However, when  $3 \times 10^7$  cells/ml are used, the bandsread is reduced from some 35-40 fractions down to 16-18 fractions. Pituitary cells also appear to be effected. Results of CFES experiments on STS-7 suggest that this change in resolution may be due to too high an ion concentration in the sample stream resulting in an ionic concentration mismatch between the sample and the carrier buffer. It may also be due to cell-cell aggregations or other cellular interactions which modify the apparent field intensity or other parameters of the electrophoresis process. This study is using the CFES ground research unit and other traditional techniques to characterize this phenomenon. When the mechanism is determined, computer projections will be made to predict whether or not these limits will be relieved when using the CFES under microgravity conditions.

This effort was initiated in 1985. The FY86 effort has measured the ionic concentrations in two buffer formulations previously used for microgravity separations of kidney cells. Hybridoma cells were studied in the same buffer and in an altered formula with reduced ionic strength. Pituitary cell separations using CFES were expanded to include flow cytometer studies to determine if cell-cell dumping could be determined in the highly concentrated sample streams.

This year, studies will continue using the CFES to perform separations at different cell concentrations to better define the threshold for the bandsread narrowing. CFES operating parameters will be altered throughout a normal range to determine if the narrowing can be counteracted. Different cell types will be tried to determine if there are great variations in threshold among various candidate cell types. Cell-cell aggregation in the sample stream will be measured, and the effective field strength in various parts of the sample stream will be modeled. Results will be used to predict changes, if any,

in the separated bandwidth when the CFES flight unit is used with its greater range of operating conditions.

## Mathematical Models of Continuous Flow Electrophoresis

Princeton University  
Dr. Dudley A. Saville  
NAS8-32614 (NASA Contact V. Fogle, MSFC)  
August 1977 - January 1986

The objective the the investigation is to develop a comprehensive understanding of how the flow and temperature fields affect the performance of continuous flow devices and develop models to describe the behavior of small particles in the sample stream.

The research is divided into two parts: the study of flow and temperature fields and the behavior of suspended particles. The first part is theoretical, the second both theoretical and experimental. In the first part flow stability, flow development, and fully developed configurations were studied, with special emphasis on the relation between flow structure and electrode configuration. A general computer simulation of the fractionation of particle mixtures in a CFE device was also constructed.

The second major focus of the work is the electrokinetic properties of suspended particles. Studies were made of the electrophoretic mobility of suspended particles in dilute and concentrated systems and the electrical conductivity of dilute concentrated suspensions. The sedimentation potential was also investigated in connection with the latter study. In these investigations we sought to establish: (1) how the mobility of the suspended particles is related to the buffer composition and the presence of nearby particles, and (2) how the bulk conductivity of a suspension is related to the buffer properties, the electrokinetic properties of the suspended particles, and the number of particles per unit volume.

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## Electrophoresis Technology

Marshall Space Flight Center  
Dr. Robert S. Snyder  
In-House

The objectives of this program are to: (1) analyze the fluid flow and particle motions during continuous flow electrophoresis by experimentation and computation; (2) characterize and optimize electrophoretic separators and their operational parameters; (3) develop innovative methods to accomplish electrophoretic separations in space; (4) analyze the electrophoretic process using apparatus that has been characterized or modified to perform in a predictable manner and according to procedures that have been developed to yield improved separation.

A new high-resolution apparatus designed for space has been built as a laboratory prototype. Using a moving wall with a low zeta potential coating, the major sources of flow distortion for an electrophoretic sample stream are removed. High resolved fractions, however, will only be produced in space because of the sensitivity of this chamber to buoyancy induced convection in the laboratory. However, preliminary laboratory experiments and analysis confirm important features of its operation.

The second and third flights of the McDonnell Douglas Astronautics Corporation continuous flow electrophoresis system carried samples developed at MSFC intended to evaluate the broad capabilities of free-flow electrophoresis in a reduced gravity environment. Biological model materials, hemoglobin and polystyrene latex microspheres, were selected because of their past use as electrophoresis standards and as visible markers for fluid flow due to electroosmosis, spacecraft acceleration or other factors. The specific objective of the experiments was to assess the dependence of the separation resolution on the properties of the sample and its suspension solution. Photographs of the space electrophoresis experiments, analysis of the collected fractions and recent laboratory experiments show the sensitivity of resolution to the electrical properties of the sample solution and electrophoresis curtain buffer.

A small electrophoresis chamber, 15 cm x 5 cm x 0.5 cm has been built for observing the sample stream behavior along the three orthogonal axes. This chamber has been assembled with the associated pumps, fluid metering and power supplies to observe sample deflections under various experimental conditions such as high electric field and reduced electroosmosis. This instrument will be used to generate data for the various models of electrophoresis now available.

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Herren, B. J., Shafer, S. C., Van Alstine, J. M., Harris, J. M., and Snyder, R. S., "Control of Electroosmosis in Coated Quartz Capillaries," J. Colloid & Interface Sci., 1985 (submitted).

## 5. GLASSES AND CERAMICS

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## Multimode Acoustic Research

Jet Propulsion Laboratory

Dr. Martin Barmatz

NAS7-918

October 1, 1981 - September 30, 1986

There is a recognized need for high temperature containerless processing facilities that can efficiently position and manipulate molten samples in the reduced gravity environment of space. The primary objectives of this task are to develop theoretical models of new classes of acoustic levitation and provide experimental validation of these models using research levitation devices.

The ultimate goals of this research is to develop sophisticated high temperature manipulation capabilities such as selection of arbitrary axes of rotation and rapid sample cooling. This program will investigate new classes of levitation in rectangular, cylindrical and spherical geometries. The program tasks include calculating theoretical expressions of the acoustic forces in these geometries for the excitation of up to three acoustic modes (multimodes). These calculations are used to (1) determine those acoustic modes that produce stable levitation, (2) isolate the levitation and rotation capabilities to produce more than one axis of rotation, and (3) develop methods to translate samples down long tube cylindrical chambers. Experimental levitators will then be constructed to verify the stable levitation and rotation predictions of the models.

Theoretical analyses carried out under this task have predicted stable acoustic levitation in rectangular, cylindrical and spherical resonators using one acoustic mode of excitation. This theory is now being used to design cylindrical single mode levitators for use at very high temperatures ( $\geq 1500^{\circ}\text{C}$ ). The translational and rotational levitation stability of these single mode levitators are now being evaluated at elevated temperatures.

### Publications

Barmatz, M., "A New Method for Acoustic Containerless Processing of Materials," in Proceedings of IEEE 1984 Ultrasonics Symposium, Volume 2, 1984, p. 436.

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Solid-State Combustion Synthesis of Ceramics and Alloys in  
Reduced Gravity

Los Alamos National Laboratory  
Dr. Robert G. Behrens  
Dr. Steven M. Valone  
C-80011-F (NASA Contact: Janet Hurst, LeRC)

The objective of this research study is to demonstrate the role of gravity in the formation of ceramics and alloys by combustion synthesis.

Examine the reactive fluid flow aspects of condensed phase combustion processes at microscopic and atomic levels through wetting experiments and numerical simulations.

Publications

Valone, S. M., "Reactive Transport at Ti + C Interfaces," in Proceedings of the DARPA/Army Symposium on Self-Propagating, High-Temperature Synthesis,

Behrens, R. G. and Brush, H. T., "Thermochemical Modeling of Ceramic Systems Formed by Combustion," in Proceedings of DARPA/Army Symposium on Self-Propagating, High-Temperature Synthesis.

## Glass Formation in Reluctant Glass Formers

Marshall Space Flight Center  
Dr. Edwin C. Ethridge  
In-House

The objective of this research is to investigate the crystallization kinetics and glass forming ability of reluctant glass formers. This could ultimately aid the formation of bulk samples of unique glass compositions outside of normal glass forming regions allowing the optimization of certain properties of the glass.

One important aspect of processing in space is the containerless undercooling of molten substances. Theoretically, the extent of undercooling can be greatly enhanced by solidifying in the absence of heterogeneous nucleation resulting from contact with crucibles or molds. The containerless solidification of reluctant glass formers may permit much slower cooling rates to form glasses than is otherwise required.

This work has concentrated on establishing techniques for the measurement of crystallization kinetics and critical cooling rates. The glass formation ability and crystallization kinetics of  $\text{Ga}_2\text{O}-\text{CaO}$  and several  $\text{Al}_2\text{O}_3-\text{CaO}$  compositions have been measured. An apparatus has been set up to measure the temperature of spherical samples on a thermocouple at large cooling rates. The time and temperature of nucleation is recorded and the probability of nucleation at various cooling rates can be measured. From this curve, it has been defined that the critical cooling rate to form a glass is that cooling rate at which there is a 95% probability of forming a glass.

Other techniques being developed include superimposing processing variable (temperature) from a fast data acquisition system onto the video from the microscopic examination of sample on a strip heater furnace. From this apparatus, crystal growth rates at various undercooled temperatures are being measured.

Another apparatus under development is an airjet levitator with heating from a 700 watt  $\text{CO}_2$  laser. This has been used to levitate molten  $\text{Al}_2\text{O}_3$  to temperatures as high as  $2800^\circ$  and resolidify containerlessly. The plan is to utilize this apparatus to investigate the limit of undercooling in the absence of heterogeneous nucleation.

## Publications

Ethridge, E. C., Curreri, P. A., and Pline, D., "Glass Formation Studies in  $\text{Ga}_2\text{O}_3$ -CaO and  $\text{Al}_2\text{O}_3$ -CaO Systems," J. Am. Ceram. Soc., 1985 (submitted).

## Study of Foaming in Glass Melts under Microgravity

Case Western Reserve University

Dr. Pavel Hrma

NAG3-740 (NASA Contact: J. Hurst, LeRC)

October 1, 1986 - October 31, 1987

The objective of this research is to study the effect of microgravity on formation and destruction of foams in high viscosity melts. Under the conditions of reduced gravity surface forces will become dominant in determining foam structure, the mode of foam destruction, and the foaming and foam collapsing rates. This will allow us to treat buoyancy and capillarity contribution separately, and thus the theoretical understanding of foaming will be enhanced.

Foaming of aqueous and nonaqueous liquids and high-temperature melts is common in industry as well as in nature. Foam formation and foam destruction are often jointly controlled by surface forces and gravity, especially if the foaming material is a glass or a slag. The use of microgravity to study foaming in glass melts will enable a separation of these two forces, that is, buoyancy and capillarity, which is otherwise very difficult or impossible: surface forces can be affected by experimental conditions only to a very limited extent. If the gravity driven viscous drainage is removed, many practical problems of foaming can be clarified. For example the mechanism of the effect of foam stabilizers (like of combination of  $Al_2O_3$  and  $Na_2SO_4$ ) and destabilizers (reducing atmosphere, carbon) or the mechanism of reboil bubble nucleation.

Glass as a foaming material is preferable for its technological significance as well as for its technical advantages. Glass or slag foaming is a common side effect in glassmaking and metal processing, and solid glass foams have a wide range of practical applications.

Studying of foaming under microgravity will enhance understanding of the role of buoyancy in foam making and foam destruction on Earth. Any improvement in glassmaking or foam making is highly desirable because of the large scale at which these processes are performed.

With buoyancy forces substantially reduced, capillarity will dominate foaming, and forces determining structure, stability, evolution and collapse of foams can be better understood. If gravity driven drainage is removed, the mechanism of foam stabilization and destabilization can be clarified. If bubble motion driven by body force is stopped, phenomena connected with bubble production, such as reboil, can be comfortably approached.

## Levitation Studies of High Temperature Materials

Rice University

Professor John L. Margrave

NAS8-33199 (NASA Contact: L.B. Gardner, MSFC)

December 1983 - December 1986

This research is a proposed three-year program which is designed to expand capabilities for doing levitation research by moving into the microgravity of space. It will allow the establishment of highly reliable thermodynamic and other properties of elements like silicon and boron in both solid and liquid states, without the risk of container contamination. Also, the phenomenon of super-cooling, nucleation and kinetics of crystal growth which are so important in semiconductor development can be studied without the interference of gravity, vibrations, container impurities and dust. Studies will be conducted which yield monochromatic spectral and hemispherical emissivities of liquid boron and liquid silicon at various wavelengths and temperatures. Also, the densities of the liquids will be determined by a photographic technique at various temperatures.

At Rice University levitation and melting of multigram quantities of pure metals in carefully designed coils coupled with a radio-frequency induction heater has been accomplished. Thus far studies of levitated liquid metals at high temperatures have shown: (1) that the heat capacities of most liquid metals are greater than those of the solids at the melting point; (2) that the heat capacities and emissivities of most liquid metals are constant for several hundred degrees above the melting points; and (3) that deviations from linearity do occur for a few refractory metals at temperatures above 3000 K, consistent with predictions based on levitation and exploding wire studies.

Research planned includes: (1) complete studies of liquid aluminum. Further studies are needed before definitive thermodynamic properties can be provided; (2) measure densities of liquid transition metals using photographic techniques as a  $f(T)$  from MP to MP + 500 degrees, or higher if possible; (3) measure  $\epsilon(\lambda, T)$  for liquid metals; (4) initiate electromagnetic levitation studies of elemental Hf; and (5) work with Dr. R.T. Frost of General Electric and Dr. D.W. Bonnell of NBS to design a chamber for levitation studies of silicon and boron in a space shuttle experiment.

Containerless High Temperature Property Measurements by Atomic Fluorescence

Midwest Research Institute  
Dr. Paul C. Nordine  
NAG8-465 (NASA Contact: L. Gardner, MSFC)  
June 1981 - December 1986

Laser induced fluorescence (LIF) is used this program to develop and test new methods for containerless high temperature property measurements and process control. High temperatures are achieved in containerless experiments by use of electromagnetic (EM) and/or CW CO<sub>2</sub> laser heating with aerodynamic and/or EM levitation of specimens. The capabilities and limitations of earth-based experiments are demonstrated and the information necessary to design improved experiments in space is obtained.

LIF studies of Al<sub>2</sub>O<sub>3</sub>, W, Mo, and LaB<sub>6</sub> evaporation and of supersonic levitation jet structure were carried out. Methods for specimen temperature measurement by LIF were evaluated. The results include accurate absolute vapor pressure measurements on levitated spheres of LaB<sub>6</sub>, a material that reacts with all of the container materials used in previously reported work. Also, accurate spectral absorption coefficient measurements were obtained on sapphire at  $\lambda = 0.66 \text{ m}$  and  $T = 1800 \text{ to } 2327\text{K}$ , from measured vapor pressure versus apparent temperature and the known relation between vapor pressure and true temperature.

A new program was initiated to use LIF to measure component activities on liquid alloys, and for process control in containerless synthesis experiments. Liquid specimen emissivity and apparent temperature are simultaneously measured with a polarizing emissivity meter and an optical pyrometer. Liquid alloy component activities are obtained from the ratio of vapor species LIF intensities for alloys and pure component elements. Also, two laser LIF experiments measure vapor velocity or electronic state distributions from which vapor temperatures may be calculated.

Publications

Nordine, P. C. and Schiffman, R. A., "Containerless Laser Induced Fluorescence Study of LaB<sub>6</sub> Evaporation," High Temp. Sci. **20**, 1-20 (1985).

Nordine, P. C., "The Accuracy of Multi-Color Optical Pyrometry," High Temp. Sci., 1985 (accepted).

## The Formation of Ordered Microstructures by Slip Casting and Related Processes

Princeton University

Dr. W. B. Russel

NAG3-584 (NASA Contact: Doug Kiser, LeRC)

November 15, 1984 - November 14, 1985

This research aims to develop a fundamental understanding of the phenomena controlling the rate of formation and morphology of the dense phase produced in processes such as sedimentation, ultrafiltration, and slip casting. These processes are widely used to concentrate small particles from dilute liquid dispersions, and the important role of the structure of the sediment/filter cake/casting on the performance of the process and the quality of the product is generally recognized. Nonetheless the effect of process parameters and the interparticle forces on the morphology remains ill-defined.

The first step in a macroscopic model, based on a conversion equation for the particles including the fluxes due to convection, gravity, and diffusion. The sedimentation and diffusion coefficients link the fluxes to the local volume fraction and the interparticle potential. The model extends significantly beyond those currently available by including both the dispersed and dense phases, thereby supplying a complete description of the process. Solutions determine the volume fraction as a function of position and time, providing the basis for subsequent description of the degree of ordering in the sediment/filter cake/casting.

Monodisperse silica spheres in cyclohexane have been synthesized for experiments to test the validity of the theory. These are known to behave as hard spheres and to be nearly transparent, enabling scattering experiments. The theory and experiments represent steps toward an understanding of the conditions for which hard sphere suspensions form ordered dense phases.



## Bulk Formation of Metallic Glasses and Amorphous Silicon from the Melt

Harvard University

Dr. Frans Spaepen

NAS8-35416 (NASA Contact: L. Gardner, MSFC)

May 16, 1983 - May 15, 1986

The objectives of this research are: (1) to find new procedures and compositions for producing metallic glasses in bulk at slow cooling rates; (2) to attempt to form the amorphous phase of the tetrahedrally coordinated elements (Si and Ge) by undercooling the melt; and (3) to examine the crystal nucleation behavior to pure liquids and glass formers experimentally and theoretically.

By exposing a metallic glass forming melt for an extended period of time to a surrounding molten oxide flux, heterogeneous nucleants (primarily at the surface) can be eliminated. By using metallic glass compositions with a high relative glass transition temperature ( $T_g/T_l > 0.6$ ), such as  $Pd_{40}Ni_{40}P_{20}$ , homogeneous nucleation also becomes negligible. We have succeeded in obtaining large (5g) masses of this alloy, using a molten  $B_2O_3$  flux. This procedure is more effective and more reproducible than the vacuum treatments that were developed earlier. It also allows us to reheat the glass to the liquid state without crystallization, and hence to study the physical properties of the liquid over the entire metastable range from  $T_g$  to  $T_l$ .

We are also studying the undercooling of liquid Si and Ge droplets (0.5 - 1mm) by drop tubes, fluxing and vacuum treatment techniques, to explore the possibility of forming amorphous Si or Ge directly by slow cooling below the melting point,  $T_{a1}$ , of the amorphous phase. Uncoated Si droplets in vacuum have been undercooled by 250°C. Ge droplets coated with a molten  $B_2O_3$  flux have been undercooled by 280°C. In both cases, the solidified samples were polycrystalline, and the temperature at which nucleation was observed was at or below  $T_{a1}$ .

An upper limit to the homogeneous crystal nucleation frequency, at the maximum undercooling, of  $2 \times 10^4/cm^3 \cdot s$  and  $8 \times 10^6/cm^3 \cdot s$  was determined for Si and Ge, respectively.

### Publications

Kui, H. W., Greer, A. L., and Turnbull, D., "Formation of Bulk Metallic Glass by Fluxing," Appl. Phys. Lett. **45**, 615 (1984).

Devaud, G. and Turnbull, D., "Undercooling of Molten Silicon," Appl. Phys. Lett. 46, 844 (1985).

Kui, H. W. and Turnbull, D., "The Melting of Ni<sub>40</sub>Pd<sub>40</sub>P<sub>20</sub> Glass," Appl. Phys. Lett. 47, 796 (1985).

Devaud, G. and Turnbull, D., "Undercooling of Liquid Germanium," in Materials Research Society Proceedings 57, 1986 (in press).

## Glass Research

Jet Propulsion Laboratory  
Dr. Michael C. Weinberg  
In-Center

Research efforts span three general areas of glass science: glass refining, gel-derived glasses, and nucleation and crystallization of glasses. Gas bubbles which are present in a glass product are defects which may render the glass totally useless for the end application. For example, optical glasses, laser host glasses, and a variety of other specialty glasses must be prepared virtually defect free to be employable. Since a major mechanism of bubble removal, buoyant rise, is virtually inoperative in microgravity, glass fining will be especially difficult in space. On the other hand, the suppression of buoyant rise and the ability to perform containerless melting experiments in space allows the opportunity to carry our several unique bubble experiments in space. Gas bubble dissolution studies may be performed at elevated temperatures for large bubbles with negligible bubble motion. Also, bubble nucleation studies may be performed without the disturbing feature of heterogeneous bubble nucleation at the platinum walls. Currently, ground based research efforts are being performed in support of these potential flight experiments.

Gel precursors can be used to prepare unique metastable glasses and glass ceramics. They offer several special advantages for glass preparation in a containerless experiment in space in that they can be prepared as amorphous, homogeneous materials in an ultrapure state. However, there is evidence that the structure and phase transformation behavior of these materials may be different from the corresponding glasses prepared by conventional glasses. Since these differences can adversely affect optical, mechanical, and other properties, there is need to explore the extent and origins of these differences. Our ground-based research efforts have aimed to elucidate in which systems such differences may be expected to occur and their causes.

The ability to prepare inorganic oxide glasses may be limited by the tendency of the melt to heterogeneously nucleate and crystallize from the container wall. Also, the ability to study free surface crystallization may be impaired by crystallization events initiating at the melt-container interface. In space such events may be avoided through the use of containerless processing. We have aimed to seek simple systems which exhibit a tendency to

crystallize at the melt-container interface and to study their ground based crystallization behavior.

### Publications

Weinberg, M. C., Neilson, G. F., and Uhlmann, D. R., "Homogeneous Versus Heterogeneous Crystal Nucleation in  $\text{Li}_2\text{O} \cdot 2 \text{SiO}_2$  Glass," J. Non-cryst. Solids **68**, 115 (1984).

Hornyak, E. J. and Weinberg, M. C., "Velocity of a Freely Rising Gas Bubble in a Soda-Lime-Silicate Glassmelt," J. Am. Ceram. Soc. **67**, C-244 (1984).

Weinberg, M. C., "Finite Sample Size Effects in Transformation Kinetics," J. Non-cryst. Solids **72**, 301 (1985).

Weinberg, M. C. and Neilson, G. F., "On the Possibility of Critical Radius Measurements in the Homogeneous Crystal Nucleation of Glass," J. Non-cryst. Solids **74**, 177 (1985).

Neilson, G. F., Smith, G. L., and Weinberg, M. C., "The Effect of Chloride Incorporation on the Crystallization of a ZBLA Fluoride Glass," J. Am. Ceram. Soc. **68**, 629 (1985).

Neilson, G. F., Weinberg, M. C., and Smith, G. L., "Effect of OH Content on Phase Separation Behavior of Soda-Silica Glasses," J. Non-cryst. Solids **82**, 137 (1986).

Weinberg, M. C., "On the Location of the Maximum Homogeneous Crystal Nucleation Temperature," J. Non-cryst. Solids **83**, 98 (1986).

Weinberg, M. C., Smith, G. L., and Neilson, G. F., "Glass Formation and Crystallization of High Lead Content  $\text{PbO-B}_2\text{O}_3$  Compositions," Bull. Am. Ceram. Soc., 1986 (in press).

Weinberg, M. C., "The Crystallization of Small Particles and Droplets," J. Non-cryst. Solids, 1986 (in press).

Weinberg, M. C. and Neilson, G. F., "A Comparison of the Phase Transformation Behavior of Gel-Derived and Ordinary  $\text{Na}_2\text{O-SiO}_2$  Glasses," in Sol-Gel Technology (L. Klein, ed.), 1986 (accepted).

Weinberg, M. C., "On the Possibility of Diffusionally Driven Oscillations in Two Component Gas Bubbles," Chem. Engr. Sci., 1986 (accepted).

Weinberg, M. C., "Physical Data Measurements and Mathematical Modelling of Simple Gas Bubble Experiments in Glassmelts," J. Non-cryst. Solids, 1986 (accepted).

## High-Temperature Controlled Redox Crystallization Studies

Johnson Space Center  
Dr. Richard J. Williams  
In-House

Many silicate compositions crystallize dense phases, which settle during ground-based experiments; and thus gravity can influence the texture and the chemical evolution of the crystallizing system.

For complex silicates containing redox sensitive ions (e.g., Fe, Ti, Cr) crystallization must be performed under controlled and known redox conditions. The conventional gas-mixing techniques used in ground-based studies require large quantities of flowing gases and are not compatible with flight. The purpose of this investigation has been to develop a test system in which controlled redox experiments can be performed in the microgravity environment and to exploration of energy melting and crystallization phenomena for subsequent space experimentation.

A laboratory prototype system has been designed, built, and tested; the system uses a solid-ceramic electrolyte cell to control, measure, and manipulate the oxygen fugacity during high temperature experimentation. The redox conditions are produced and maintained by using the ceramic electrolyte to electrolyze the gas in contact with the sample. The system operates well at temperatures between 1000° and 1400°C and at oxygen fugacities between those of the Ni-NiO and Fe-FeO reactions. Control of the temperature, oxygen fugacity, and the rate of change of these variables with time is provided by a personal computer; accuracies are the same as those obtained by conventional techniques. The system totally recycles all gases and thus is directly adaptable for flight. A proposal for flight development has been submitted.

### Publications

Williams, R. J., Lofgren, G. E., Jolly, G. D., Lanier, A. B., and Carter, W. I., "A System for Control and Manipulation of Redox Conditions in Microgravity Experiments," J. Metals 37, 10 (1985). (Abstract)

**6. COMBUSTION SCIENCES**

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Design and Evaluation of an Apparatus for Experiments on the Vaporization of Fuel Droplets in a Supercritical Environment at Microgravity Conditions

University of Wisconsin-Madison  
Professor Gary Borman  
Professor P. S. Myers  
P. V. Farrell, General Motors Research Lab  
Dr. Bruce Peters, General Motors Research Lab  
NAG3-718 (NASA Contact: Kurt Sacksteder, LeRC)  
April 15, 1986 - April 14, 1987

This program represents a joint venture between the University of Wisconsin-Madison and General Motors Research Laboratories. The study concentrates on the details of fuel spray breakup and vaporization in conditions similar to those of a direct injection diesel engine. The environment into which fuel is typically injected is well above the fuel critical pressure and may be above fuel critical temperature. Near-critical property and surface tension effects are known to vary rapidly as the critical point is approached, possibly leading to unexpected droplet breakup and vaporization effects. In order to study these effects in detail, it is desired to eliminate all convective effects (convective heat and mass transfer). One way to eliminate natural convection in a non-isothermal, multi-component field with diffusion is to reduce acceleration by operating at very small (microgravity) levels.

The current grant funds the first year of a planned three year program. The objectives of this program are: (1) to evaluate the possibilities and the likely constraints of conducting droplet vaporization experiments at supercritical conditions in microgravity; (2) to design experiments and apparatus to allow microgravity experimentation; and (3) to perform experiments to study the behavior of liquid droplets in supercritical microgravity environments.

To accomplish the first objective, which is the major goal of this first year of the program, a series of tasks will be undertaken. These tasks include: selection of candidate liquids and candidate surrounding gases based on their thermodynamic and diffusion properties; sensitivity analysis of near critical fluid properties for a variety of liquid-gas pairs using a numerical solution to an unsteady droplet diffusion-vaporization model; design and construction of a droplet generating rig, most likely based on a current NASA design; initial planning for generation of the supercritical temperature and pressure field required; fluid flow modeling of candidate pressurization and heating techniques; and selection of an overall configuration for the compression rig.

Subsequent years of the program will accomplish objectives (2) and (3). These years will concentrate on construction of the test rig, development of optical instrumentation techniques for temperature and concentration measurements, integration of all systems, and finally testing of the apparatus and actual performance of the experiments.

#### Publications

Farrell, P. V. and Peters, B. D., "Droplet Vaporization in Supercritical Pressure Environments," Acta Astron., 1986 (in press).



## The Effect of Gravity on Premixed Turbulent Flames

University of California, San Diego  
Professor Paul A. Libby  
NAG3-654 (NASA Contact: Kurt Sacksteder, LeRC)  
September 1, 1985 - December 31, 1986

Recent experimental and theoretical research has shown that the interaction of force fields arising from gradients of either mean pressure or Reynolds shear stresses with density fluctuations due to heat release in premixed turbulent flames leads to new mechanisms of turbulent transport and turbulence production. Gravity provides an additional force field which may result in significantly different behavior for such flames propagating upward and downward.

A combined theoretical and experimental effort is underway. The theoretical analysis involves extension of existing theories of premixed turbulent flames to include the influence of gravity. Preliminary work indicates that a model for the mean rate of creation of product is required in this extension whereas in previous analyses such a model could be avoided by a change of independent variables. Attention is presently focused on the examination of a suitable description of this chemical term. The experimental work is getting underway and will involve preliminary experiments in a vertical tube filled with reactants to determine the extent of the influence of gravity on turbulent flames by comparing upward and downward propagating behavior.

### Publications

Hamins, A., Heitor, M., and Libby, P. A., "Gravitational Effects on the Structure and Propagation of Premixed Flames," Acta Astron., 1986 (submitted).

Time-Dependent Computational Studies of Flames in Microgravity

Naval Research Laboratory  
Dr. Elaine S. Oran  
Dr. K. Kailasanath  
(NASA Contact: K. Sacksteder, LeRC)

The objective of this research is to: (1) quantify the differences between flame initiation, propagation and quenching in zero and finite gravity, and (2) to investigate the basic mechanisms controlling flame ignition, propagation and quenching.

The focal issues to be addressed are: (1) will a mixture which is not flammable on earth burn in a reduced gravity environment? (2) Are the minimum ignition energies different for mixtures which are flammable both at normal and reduced gravity? and (3) Are there differences in the propagation of flames in normal and reduced gravity? The approach is to use detailed time-dependent, one- and two-dimensional numerical models to calculate flame properties and then to look for a quantitative comparison between the predictions and experiments on earth and in space. These models solve the multispecies coupled partial differential reactive flow equations. These models contain detailed chemical kinetics mechanisms, algorithms for thermal conduction, molecular and thermal diffusion, and convective transport and include the effects of gravity. The results will be correlated with the experiments of P. Ronney, R.A. Strehlow, and A. Berlad who are currently involved in the NASA microgravity combustion program.

A Fundamental Study of Smoldering with Emphasis on  
Experimental Design for Zero-G

University of California, Berkeley  
Professor Patrick J. Pagni  
Professor A. Carlos Fernandez-Pello  
NAG3-443 (NASA Contact: Dr. K. Sacksteder, LeRC)  
October 1984 - December 1986

The objective of this project is to identify key sets of low-gravity experiments which would critically compliment a larger set of more easily performed normal-gravity experiments to elucidate the phenomena found in smoldering combustion.

It is planned to follow through on the conceptual design of these experiments by participating in the future in the fabrication of the refined apparatus and in the actual data collection and interpretation. Low-gravity experiments are appropriate for smoldering combustion because of the complexity of smoldering which requires every means possible to discriminate among the many chemical and physical mechanisms operative in most smoldering combustion scenarios. Efforts will be primarily analytical, attempting to identify appropriate approximations and dominant dimensionless groups based on existing data and state-of-the-art combustion modelling. Transient stability questions such as ignition, extinction and choices among charring, tarring, or flaming modes will be included.

Publications

Dosanjh, S. S., Pagni, P. J., and Fernandez-Pello, A. C., "Forced Current Smoldering Combustion," in Proceedings of Twenty-First International Symposium on Combustion, Munich, August 1986 (accepted).

Peterson, J., Dosanjh, S. S., Fernandez-Pello, A. C., and Pagni, P. J., "Buoyancy Effects on Smoldering Combustion," Acta Astron., 1985 (in press).

## Ignition and Flame Spread Above Liquid Fuel Pools

University of California, Irvine  
Professor William A. Sirignano  
NAG3-627 (NASA Contact: Kurt Sacksteder, LeRC)  
March 1, 1985 - November 30, 1986

The objectives of this program are to obtain theoretical and computational results that will guide the development of an experiment on the subject of flame propagation above liquid fuel pools. The reduced gravity version of the experiment may contain the heat and mass transport aspects without including the chemical reaction and energy release. Discussions are presently underway as to whether the UCI mission should include the development of an earth-gravity experimental program.

The achievements involve the development and use of computer codes that allow the study of heat transport in an enclosed, two-phase system. Effects of buoyancy, surface-tension, radiation, wall thermal inertia, and unsteadiness in liquid and gas heating and motion are included in the analysis. A parameter study has been made and the relative importance of each of these physical effects have been determined in each segment of the parameter range. Primary parameters are Grashof number, Marangoni number, height-to-radius ratio of container, and height of liquid in the container.

### Publications

Aggarwal, S. K., Iyengar, J., and Sirignano, W. A.,  
"Enclosed Gas and Liquid with Nonuniform Heating from Above," J. Heat and Mass Transfer, 1986 (accepted).

### Presentations

Abramzon, B., Edwards, D. K., and Sirignano, W. A.,  
"Transient Natural and Surface-Tension-Driven Convection in a Two-Layer Gas-and-Liquid Enclosure with Nonuniform Radiative Transfer," presented at 24th AIAA Aerospace Sciences Meeting, Reno, Nevada, January 1986, AIAA 86-0578.

## Flammability Limits of Gases Under Low Gravity Conditions

University of Illinois, Urbana

Dr. Roger A. Strehlow

NAS3-23770 (NASA Contact: K. Sacksteder, LeRC)

December 24, 1984 - December 23, 1985

The objective of this research is to determine the effect of gravity on the flammability limits of premixed methane-air and propane-air flames in a standard 51 mm diameter flammability tube and to determine, if possible, the fluid flow associated with flame passage under different conditions.

The current phase of the study is to determine the lean flammability limit and the behavior of the flame near the limit for methane-air or propane-air mixture using a standard flammability tube in the vertical position under conditions of micro, fractional, and super gravity. This has been accomplished using the 8 tube flammability study rig in NASA's Lewis Research Center Lear Jet facility and flying the prescribed g-trajectories while the experiment is being performed. Data was recorded photographically using the visible light of the flame. The data that was acquired are the shape and propagation velocity of the flame under various g-conditions for methane or propane compositions that are inside the flammable limits and the effect of gravity on the limits. Additionally, the effect of varying gravity during flame propagation was investigated. Real time accelerometer readings for the three orthogonal directions were displayed in full view of the cameras and the framing rate of the cameras was used to measure velocities. Twenty-five flights were completed with up to 8 data points collected on any one flight. The data is currently being readied for publication in the open literature.

### Publications

Strehlow, R. A., Noe, K. A., and Wherley, B. L., "The Effect of Gravity on Premixed Flame Propagation and Extinction in a Vertical Standard Flammability Tube," in Proceedings of 21st International Symposium on Combustion, The Combustion Institute, 1985 (in press).

## 7. EXPERIMENTAL TECHNOLOGY AND GENERAL STUDIES

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## Advanced Optical Measurement Techniques

Marshall Space Flight Center  
Dr. Robert B. Owen  
Mr. William K. Witherow  
In-House  
Continuing Task

Optical measurement techniques are critical subsystems in several current and projected MPS experiments. This effort deals with the laboratory development and use of these techniques for scientific and engineering support of such experiments, as well as the development of engineering breadboards for future flight experiments. The task encompasses four areas: (1) Improved Techniques for Optical Measurement; (2) KC-135 Low Gravity Simulation Flight Preparation; (3) In-Line Holographic Microscopy; and (4) Holographic Optical Schlieren (HOSS).

The first area provides for laboratory breadboarding of optical techniques which show potential for use in MPS experiments. A color schlieren system is being built which has many of the advantages of interferometry without the vibration sensitivity and alignment requirements commonly associated with these systems. This technique appears extremely promising and is being actively investigated. The development of a fiber optics interferometer has begun.

The second area allows improved optical systems and breadboards to be built for experiments and hardware tests on the KC-135 aircraft. The improved Mach-Zehnder interferometer designed previously has been constructed and is scheduled to be flown this winter. A field color schlieren system has been built and flown using a variety of fluid samples. The system performed well under microgravity conditions, and is presently being considered as a shuttle middeck locker package.

The third area is concerned with improvements and applications of holographic microscopy. Experiments studying the dynamics of nucleation, the separation of immiscible fluids, and Ostwald ripening have been conducted. A study of Ostwald ripening is a candidate for flight in the Fluids Experiment System (FES) on IML-2, and work has begun on designing an optical system for this experiment. Other work includes studies of protein crystal growth mechanisms using schlieren techniques which will be supplemented by interferometry.

The fourth area involves use of the Holographic Optical Schlieren System (HOSS) to develop systems which have been used to reconstruct Spacelab 3 FES holograms. The excellent quality of these holograms has interested other experi-

menters in the FES. The HOSS will therefore be reconfigured to duplicate the FES and to prepare flight experiments.

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## Advanced Containerless Processing Technology

Jet Propulsion Laboratory

Dr. Taylor G. Wang

In-Center

October 1978 - continuing task

The long-range objectives of this task are to : (1) study and advance the science of contactless positioning and manipulation of a high-temperature acoustic chamber; (2) provide technical information to the Acoustics Containerless Experimental System (ACES) engineering team; and (3) develop a set of high temperature ground-based facilities for precursor material processing experiments.

The subjects to be addressed are experimental and theoretical studies of: (1) acoustic positioning and manipulation capabilities in a high temperature gradient environment (from 25°C to 900°C); (2) acoustic waveforms, harmonic contents, power transfer, sample transport and stability associated with a high temperature gradient system; (3) high temperature ground based levitation systems which will allow us to melt, process, and solidify samples without crucibles in the laboratory; (4) KC-135 and laboratory test of various acoustic geometries which may have potential applications in the Materials Processing in Space Program; and (5) provide technical information to the ACES engineering team and establish the operation conditions for ACES.

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## B. FLIGHT EXPERIMENTS

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## 1. ELECTRONIC MATERIALS

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## Compound Semiconductor Growth in Low-g Environment

NASA Langley Research Center  
Dr. A. L. Fripp  
Dr. Roger K. Crouch, NASA HQ  
In-House

This research analyzes the effects of convection on the macroscopic and microscopic homogeneity of compound semiconductors grown by the Bridgman technique. The material of primary emphasis is lead-tin-telluride which is thermodynamically unstable in a 1-g environment. Ground based experiments have involved the measurement of thermophysical properties, development of delineating etches to show interface shapes, in-situ interface measurements using radiography, theoretical modelling of thermal, velocity, and solutal fields. Other work analyzes flow visualization using transparent materials. The first space flight based on this research was flown on the D-1 mission in October 1985. Hardware anomalies resulted in temperature profiles that were not anticipated, which has complicated the data reduction in terms of growth rates; however, it is expected that analysis and publication of the flight results will be completed within the next year.

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Growth of GaAs Crystals from the Melt in a Partially  
Confined Configuration

Massachusetts Institute of Technology  
Professor Harry C. Gatos  
Dr. Jacek Lagowski  
NAS8-36604 (NASA Contact: I. C. Yates, MSFC)  
July 1, 1984 - June 30, 1986

The objective of this research is to experimentally verify advantages of zero-gravity environment for the growth of gallium arsenide from the melt utilizing a novel partially confined configuration.

The program consists of two stages: (1) ground-based research and construction of a bread-board space growth apparatus which incorporates the concept of partial confinement of the growth melt; and (2) actual space growth experiments and post-flight analysis.

Publications

Gatos, H. C., Lagowski, J., Pawlowicz, L. M., and Dabkowski, F., and Li, C-J., "Crystal Growth of GaAs in Space," in Proceedings of 5th European Symposium on Materials Sciences under Microgravity, Schloss Elmau, 1984, ESA SP-222, pp. 221-225.

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A Comparative Study of the Influence of Convection on GaAs

GTE Laboratories, Inc.

Dr. James A. Kafalas

Mark Levinson

Ben Yacobi

Brian Ditchek

Alfred Bellows

Dr. John Gustafson

NAS3-24644 (NASA Contact: Dr. R. Lauver, LeRC)

The objective of this study is to determine the effects of buoyancy driven fluid flow on the properties of melt grown GaAs crystals.

Baseline GaAs crystals grown in the convection-free environment of the Space Shuttle will be compared to crystals grown on earth under differing fluid flow conditions as determined by gradient orientation and the presence and orientation of a magnetic field. The characterization of the GaAs crystals will correlate the degree and nature of the convection with macro- and micro-segregation effects, dislocation density distribution and electronic properties. The data will be interpreted based on model calculations of the fluid flow patterns in the melt under the various growth conditions. The improved understanding of the role of convection in the growth of GaAs gained from the proposed research will contribute to the refinement of GaAs growth techniques to produce substrate material with improved homogeneity and lower dislocation densities.

## Solution Growth of Crystals in Zero-Gravity

Alabama A&M University

Dr. R. B. Lal

Dr. R. L. Kroes, MSFC

NAS8-32945

March 1978 - December 1986

The objectives of this research task are to:

(1) develop a technique for solution crystal growth in a low-gravity environment; (2) characterize the growth environment provided by an orbiting spacecraft and to determine the influence of the environment on the growth behavior; and (3) determine how growth in a low-g environment influences the properties of a resultant TGS crystal.

The research is aimed at the study of solution crystal growth for low-g environment. In a low-g environment, buoyancy-driven convection effects are greatly reduced and, thus, can study diffusion mass transport which in 1-g is masked by convective phenomena. Triglycine Sulfate (TGS) was chosen as the candidate material because TGS crystals can be grown at comparatively lower temperature (~ 45 C). It is a transparent system so that holographic techniques could be employed to study fluid properties. TGS has technological importance for infrared detectors operating at room temperature.

One of the experiments conducted on the SL-3 mission was the growth of TGS crystals from aqueous solution using the Fluids Experiment System (FES). A new technique for solution crystal growth by extracting heat at a programmed rate from the crystal through a semi-insulating sting was successfully tested on the SL-3 mission. Two TGS crystals were grown during the flight and for the first time in an flight experiment, the growth was monitored on-board as well as on the ground by a video-schlieren technique. Holograms were taken of the solution/crystal interaction during the growth process. The FES optical system worked well and the quality of the reconstructed holograms was satisfactory. The interferograms indicated some g-jitter during the mission. The crystals were analyzed for pyroelectric properties and also for normalized detectivity  $D^*$ . The results of the SL-3 experiment will be published at a later time.

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Batra, A. K., Lal, R. B., and Aggarwal, M. D., "Electrical Properties of TGS Crystals Grown by a New Technique," J. Mat. Sci. Lett. 4, 1415 (1985).



## Growth of Solid Solution Crystals

Marshall Space Flight Center  
Dr. S. L. Lehoczky  
Dr. F. R. Szofran  
In-House  
October 1977- continuing task

The major objective of this program is to determine the conditions under which single crystals of solid solutions can be grown from the melt with a high degree of chemical homogeneity. The central aim is to assess the role of gravity in the growth process and to explore the possible advantages for growth in the absence of gravity. The initial alloy system being investigated is the solid solution semiconductor  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  with x-values appropriate for infrared detector applications in the 8 to 14  $\mu\text{m}$  wavelength region. Both melt and Te-solvent growth are being considered. The study consists of an extensive ground-based experimental and theoretical research effort followed by flight experimentation where appropriate.

Experimental facilities have been established for the purification, casting, and crystal growth of solid solution alloy systems. Facilities have been also established for the metallurgical, compositional, electrical and optical characterization of the alloys. Crystals are being grown by the Bridgman-Stockbarger and Te-solvent-zone methods and are analyzed by various experimental techniques to evaluate the effects of growth conditions on the longitudinal and radial compositional variations and defect densities in the grown crystals.

Theoretical models have been developed for the temperature distribution, and the axial and radial compositional redistribution during the directional solidification of solid solution alloys. The one-dimensional model that treats the variation of the interface temperature, and the interface segregation coefficient and velocity with composition, has been successfully used to establish effective diffusion constants for the HgCdTe alloy system. The temperature and compositional dependence of the liquid and solid phase thermal diffusivities have been measured in  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  for the composition range for  $x = 0$  to  $x = 0.3$  and are being used in conjunction with the thermal models to predict temperature distributions for various solidification conditions. Phase equilibrium parameters, growth rates and thermal conditions required to obtain various growth interface shapes have been established for the system.

Other tasks currently under study include: (1) theoretical modeling of the effects of growth rate on the shape of the solidification isotherms and radial solute

redistribution; (2) design and development of flight cartridges; (3) investigation of correlations between growth parameters and crystal defects; and (4) experimental and theoretical evaluation on the effect of processing conditions on the electrical and optical properties of the alloy crystals.

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Su, C-H., Szofran, R. F., and Lehoczky, S. L., "Solidification of  $\text{HgCdTe}$ ," presented at 115th Annual Meeting of TMS-AIME, New Orleans, March 1986.

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## HgI<sub>2</sub> Crystal Growth for Nuclear Detectors

EG&G, Inc.

W. F. Schnepfle

Dr. L. van Den Berg

H-73830B (NASA Contact: W.W. Moore, MSFC)

April 19, 1984 - April 30, 1986

The objectives of this program are to obtain a benchmark quality sample grown at low-g conditions and to study vapor growth phenomena under space conditions.

Ground-based crystals show a defect structure which impairs their performance as nuclear radiation detectors. These defects may be caused by the gravitational force acting on the crystal in its weakened state at the elevated growth temperature and by irregular convection patterns in the vapor during growth.

The program will be supported by ground-based research aimed at a more detailed understanding and description of the problems associated with the crystal growth process. Mechanical strength measurements have been performed (uniaxial compression tests) which show that the crystals exhibit slip parallel to the c-planes at stresses as low as 1/2 psi. Preliminary calculations using a simple linearized model indicate the oscillating instabilities in the convection part of the vapor transport system are unlikely, even at 1-g, provided that the utmost care is taken in the preparation of the crystal growth source material.

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## Vapor Growth of Alloy-Type Semiconductor Crystals

Rensselaer Polytechnic Institute  
Professor Heribert Wiedemeier  
NAS8-32936 (NASA Contact: D. A. Schaefer, MSFC)  
March 1978 - September 1986

The present effort is part of a continuing research program directed towards the investigation of basic vapor transport phenomena and of crystal growth properties of electronic materials. The primary objectives of ground-based studies are the development and definition of optimum experimental parameters for flight experiments. The ground-based effort includes the investigation of gravity-driven convection effects on mass transport rates and on crystal morphology for different orientations of the density gradient with respect to the gravity vector, and as a function of pressure and of temperature. In addition to the experimental tasks, theoretical efforts involve the quantitative thermodynamic analysis of the systems under investigation, the computation of fluid dynamic parameters, and the consideration of other possible effects on fluid flow under vertical, stabilizing and microgravity conditions.

The specific experiments to be performed in a microgravity environment include the investigation of vapor transport and crystal growth phenomena of the GeSe-Xenon system, and of the  $\text{Hg}_{1-x}\text{Cd}_x\text{Te-Hg}_2$  system. Emphasis for the latter system is on the mass flux, on the unseeded growth of bulk crystals, and on the growth of epitaxial layers. The above experiments are performed in closed, fused silica ampoules.

The major objectives of the GeSe-Xenon experiments are the determination of absolute mass transport rates under microgravity conditions and their comparison with corresponding ground-based data, with theoretically predicted values, and with flux data of earlier space experiments. The ground-based studies for this system are concerned with the investigation of the effects of different temperature gradients and pressures on the mass flux under vertical, stabilizing conditions, with the morphological characterization of the grown crystals, and with theoretical computations of the mass flux. With several appropriately designed GeSe-Xenon experiments, a mass flux versus pressure curve in microgravity is to be developed. Another important aspect of these experiments is the investigation of the deposition pattern and growth of GeSe Crystals by sublimation in different xenon pressures under ground-based conditions and in microgravity environment.

The objectives of the  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  experiments are to determine the positive effects of microgravity on crystal growth of ternary, alloy-type materials in terms of chemical and structural microhomogeneity. Gravity-driven convection effects on mass flux and morphology of bulk crystals have been observed under ground-based conditions. Continued experimental efforts are directed towards the optimization of temperature conditions for the bulk growth of  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  crystals in microgravity environment.

The major tasks of ground-based studies of the seeded growth of  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  layers of chemical vapor transport reactions involve systematic investigations of the growth rate, morphology, homogeneity, and electrical properties of  $\text{HgCdTe}$  layers. These studies include measurements of the effects of substrate orientation relative to the density gradient, of temperature, and of transport agent pressure on the above properties. They are performed under horizontal and vertical stabilizing conditions with the goal to observe the effects of convective interferences on layer morphology and properties. The results of on-going ground-based studies are continuously evaluated and are used for the systematic modification of grown parameters with the important goal to develop and to define optimum experimental conditions for the microgravity experiments of this system.

The results of the combined experiments are of basic scientific value and of technological significance. It is expected that these experiments will contribute to our understanding of vapor transport and crystal growth processes of binary and ternary materials on earth and in space and to establish conditions for space processing applications.

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**2. SOLIDIFICATION OF METALS, ALLOYS  
AND COMPOSITES**

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## Proposed Zero Gravity Undercooling Experiments

Massachusetts Institute of Technology  
Professor Merton C. Flemings  
Dr. Yuh Shiohara  
NAG3-597 (NASA Contact: Fred Harf, LeRC)  
December 1, 1984 - February 28, 1986

Solidification of metal alloys with initial high undercoolings generally occurs with rapid recalescence due to rapid heat release of the latent heat of fusion. The rapid heating during recalescence and post solidification alters the initially solidified structures in ways that must include partial remelting, ripening, coarsening, and decomposition of the rapidly solidified primary metastable or supersaturated phases.

The space shuttle experiments will be performed in January 1986, in an Electromagnetic Levitator (EML), which was mounted on the Materials Science Laboratory (MSL) frame. The main objectives of the space experiments are: (1) to evaluate containerless melting and solidification of nickel base and/or iron base alloys with and/or without glass coatings, (2) to develop techniques for study of recalescence behavior during the solidification of undercooled melts in a microgravity environment, (3) to develop an understanding of undercooling phenomena, and (4) to develop an understanding of the microstructures so produced.

This program comprises characterization and analysis of the results of the flight experiment, and the related ground-based experimental studies (and modeling of these and the flight experiments) on undercooling, remelting, and coarsening processes during the solidification of the alloys.

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## Liquid Phase Miscibility Gap Materials

Gelles Laboratories, Inc.

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Dr. A. J. Markworth

NAS8-32952 (NASA Contact: D. Schaefer, MSFC)

April 1978 - December 1986

The overall objective of the investigation is to determine the manner in which the microstructural features of liquid-phase miscibility gap alloys develop. The results of such a determination should make it possible to control the microstructures and the resultant properties of these alloys. The long-duration low-gravity afforded by the Shuttle allows experiments supporting this research to be conducted with minimal interference from buoyancy effects and gravitationally driven convection currents.

Ground based studies have been conducted on Al-In, Cu-Pb and Te-Tl alloys to determine the effect of cooling rate, composition, and interfacial energies on the phase separation and solidification processes that influence the development of microstructure in these alloys. Both isothermal and directional cooling experiments have been conducted. Computer simulations of the behavior of these systems under various conditions and theoretical models have supplemented the laboratory work. The ground based activities have been used as a technological base from which flight experiments have been formulated and to which such flight experiments have been compared and judged.

Four flights have been conducted aboard the Shuttle on STS-7 in the Materials Experiments Apparatus (MEA) during June 1983. Two of these experiments involve aluminum-indium alloys which have been processed isothermally in a configuration that avoids the presence of a free melt surface and thus eliminates a major source of convection, namely that arising from atom gradients in surface tension along this free surface. A third experiment, also involving an aluminum-indium alloy, is concerned with thermocapillary induced droplet migration. In this experiment, the alloy after homogenization has been cooled to ambient under a controlled temperature gradient. The fourth experiment deals with the melting and solidification of two tellurium-thallium liquid phase miscibility gap alloys. This last experiment is aimed at understanding the influence of interfacial energy and droplet concentration on the phase separation process.

The MEA/Al experiments described above have for the most part been carried out successfully and much valuable information and insight has been obtained. The example, from the results of the isothermal experiments on the

aluminum-indium alloys, it can be concluded that the convective flows arising from a free surface make a small contribution to the coalescence process relative to other processes which may be occurring during phase separation. The gradient cool experiment has also produced noteworthy results in that the aluminum-rich droplets produced during the phase separation appear to have moved to the cooler end of the sample rather than the hot end as predicted by theory. Plans to check this unusual result have been formulated.

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## Isothermal Dendrite Growth Experiment

Rensselaer Polytechnic Institute  
Professor Martin E. Glicksman  
NAG3-333 (NASA Contact: E. Winsa, LeRC)  
October 1983 - October 1987

The objective of this flight experiments is to assess the influence of gravity on the growth kinetics and solidification morphology of freely growing dendrites.

Dendritic growth is often associated with diffusional processes, such as heat conduction and solute flow under chemical gradients. The presence of gravity also induces buoyancy driven flows in the neighborhood of the dendritic interface, which is surrounded by chemical and/or thermal gradients and their associated density gradients. The gravity-induced convection introduces substantial changes in the heat and mass redistribution processes, which modify, in turn, the speed and shape of the dendrite. Such changes are responsible for alterations in the degree and scale of the microsegregation as well as the alignment, texture, and defect content of the solidified metal. Insofar as most metals and alloys are cast or solidified at some stage of their processing history, it is important to understand how dendrites are influenced by gravity, and how solidification may be altered in a microgravity environment. In addition, from a purely scientific standpoint, dendritic growth represents a classical non-linear pattern-forming system. Such systems have become of increasing scientific interest as the nature of their non-linear dynamics have been explored more deeply. Dendrites represent an especially simple, yet important form of dynamical pattern formation.

The Isothermal Dendrite Growth Experiment (IDGE) is a flight oriented microgravity program which will provide well-characterized material, temperature, and gravitational spectrum for observing the kinetics (growth speed) and morphology (growth form) of succinonitrile (SCN) dendrites under a variety of supercooling conditions. SCN is an especially convenient material for study of its solidification characteristics because of its transparency, melting point, and ability to be prepared in high states of purity. Moreover, SCN is body-centered cubic (BCC), enabling a direct correspondence to be made between its behavior and that of the cubic engineering metals (Fe, Al, Cu, etc.)

Three flights are planned in which the IDGE apparatus will be integrated aboard the STS on an MSL carrier. The IDGE itself is a precision stirred fluid thermostat accompanied by an integrated optical system for photographing the space and position of the solid-liquid interface. Velocity, shape, and temperature (supercooling)

will be measured to sufficient precision and accuracy to yield a valid comparison with terrestrial dendrite measurements under normal gravity. Ten supercoolings in the range of 0.1-1.0 K will be selected to perform twenty solidification events. Generally, the disparity between pure diffusional dendritic crystal growth and mixed convecto-diffusion dendritic growth increases as the supercooling decreases. On earth the differences become appreciable below about 1 K supercooling, and, consequently, the required precision in velocity and shape measurements demand a thermostat control accuracy of + 0.5 mK, which sets a hard requirement on the IDGE on-orbit performance. The photographic system will be designed as a modified shadowgraph technique to enhance depth of field and resolution of the solid-liquid profiles.

The first two flights will concentrate on the solidification of SCN, the properties of which are well established. The third flight will be designed to investigate the solidification of pivalic acid (PVA), a face-centered cubic material that exhibits a high anisotropy of the solid-liquid interfacial energy. Anisotropy has become a major scientific issue in determining the pattern forming tendencies of dendritic systems, and clearly requires careful experimental scrutiny under microgravity conditions where convection effects can be identified.

Apparatus development for IDGE is currently at the preliminary design stage. Rensselaer Polytechnic Institute is primarily responsible for overall scientific supervision as well as for the hardware associated with the dendritic growth flight chamber. Lewis Research Center has program management responsibility including overall engineering oversight and hardware development and systems integration with MSL.

Ground-based studies are also being continued to support flight hardware concepts, e.g. materials compatibility studies to assist growth chamber construction suitable for SCN and, ultimately,, PVA. Studies connected with data analysis and reduction are also on-going to assist the flight program.

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## Orbital Processing of Aligned Magnetic Composites

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NAS8-35483 (NASA Contact: F. Reeves, MSFC)  
September 1983 - March 1987

The objectives of this program are to: (1) identify and quantitatively evaluate the influences of gravitationally driven thermal-solutal convection on contained plane front solidification of binary eutectic, off-eutectic, and peritectic magnetic composites; (2) to evaluate the effectiveness of micro-g processing as a means of damping thermosolutal convection; and (3) to evaluate the uniqueness of micro-g processing relative to the best means of terrestrial convection damping.

Three flight experiments have been planned in which aligned, two-phase, magnetic composites will be grown by plane front directional solidification. Each experiment sequentially processes four independent samples in Automated Directional Solidification Furnace (ADSF) Systems. The ADSF systems use the Bridgman-Stockbarger plane front directional solidification technique. This consists of translating a thermal gradient at a programmed velocity down the length of a stationary sample (directional) under thermal conditions such that the solidification interface is flat (plane front solidification) and at a constant solidification velocity.

The first of these experiments was conducted in the Low Temperature Automated Directional Solidification Furnace System (ADSF-1), which was integrated into the mid-deck of Space Shuttle "Discovery" on Mission 51-G. The second of these experiments flew in the High Temperature Automated Directional Solidification Furnace System (ADSF-2) on the Material Science Laboratory Carrier (MSL-2) in the payload bay of the Space Shuttle "Columbia" on Mission 61-C. The 51-G experiments studied off-eutectic Bi-Mn directional solidification whereas the 61-C experiments studied Co-Sm eutectic and peritectic solidification.

The relationships between the gravity vector, heat transfer, level of thermo-solutal convection and solidification processing parameters are being studied terrestrially by varying the orientation of the gravity vector during solidification processing and by employing in-situ thermal measurement and interface demarcation techniques. These experimental results are compared with existent models of: heat flow, eutectic solidification, and off-eutectic solidification. In addition, a thermal model for the Bridgman-Stockbarger solidification technique including sample, ampoule, and translation, has been developed, and solidification models for eutectic, off-

eutectic, and peritectic solidification with partial mixing in the melt, have been derived. The level of natural thermo-solutal convection is varied by employing magnetic field (transverse and longitudinal) damping (MFD). These studies and comparative analyses with magnetically damped and micro-g processed samples will identify the role of gravitationally driven convection in plane front solidification using the Bridgman-Stockbarger technique.

Relationships between solidification processing parameters (including gravity vector), microstructure, macrostructure, chemistry as a function of fraction solidified, crystal structures, and magnetic properties are also being developed. Microstructure and macrostructure are being quantitatively analyzed using quantitative metallographic analysis techniques. Chemistries are being determined using chemical spectrophotometric absorbance, x-ray fluorescence, magnetic and microprobe analyses. Crystallography is being studied using x-ray diffraction. The magnetic measurements, which are sensitive to all of the above parameters, are used as a structure and processing-sensitive means to determine the impact of gravitationally driven convection and convective heat transfer on an important physical property.

Micro-g results from Mission 51-G have shown that diffusion controlled growth ( $k_{eff} = 1$ ) can be achieved in orbit that appear to be unachievable terrestrially, even using MFD. The results, however, showed a greatly enhanced record of Soret Diffusion in micro-g that was shrouded terrestrially by convection. Preliminary results from Mission 61-C have shown reduced contamination from the crucible walls in this reactive system, an absence of porosity, and a morphological transition from single crystal to dendritic growth that is being evaluated as described above. Two additional flight experiments have been proposed, based on these results.

The one-g experimental studies, in conjunction with theoretical analyses, will serve as a comparative base from which to evaluate the effectiveness of micro-g processing as a means of achieving diffusion controlled growth of eutectic, off-eutectic, and peritectic composites. Comparative analyses between the micro-g processed and the one-g magnetically damped results will determine the uniqueness of the orbital processing.

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## Solidification Fundamentals

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Professor John F. Wallace  
NAG3-417 (NASA Contact: Hugh Gray, LeRC)  
March 1983 - continuing task

The objective of this research is to obtain a fundamental understanding of the role of gravity during solidification of metals and alloys.

A space shuttle experiment employing the General Purpose Rocket Furnace (GPRF) in its isothermal mode of operation is planned for mid 1986. The objective of this experiment is to investigate the segregation behavior (macro and micro) in a slowly cooled sample of a binary Pb-15% Sn alloy under the influence of a greatly reduced gravitational field. Final approval for this experiment was obtained in October 1984.

Ground-based experimental work on Pb-15% Sn alloys, employing the single-cavity simulator of the GPRF at the Marshall Space Flight Center (MSFC), has been conducted as a precursor to the microgravity experiment(s). Detailed metallographic analyses and characterization of seven (7) tests conducted at MSFC is now complete. The flight test parameters are now being defined on the basis of these results. Engineering design and testing of the flight cartridge/specimen has been completed and flight specimens have been delivered to MSFC.

The above experimental research is also the subject of the M.S. thesis work of Mr. Anthony Studer. Another M.S. thesis, by Mr. Li Wang, investigated macrosegregation behavior in both Pb-15% Sn alloy and Sn-15% Pb alloys over a much wider range of cooling rates and thermal gradients than conducted at MSFC, in the single-cavity simulator.

During the past year, theoretical work on a simple model describing solidification of an array of dendrites in a binary alloy melt has also been completed. This model is based on a modification of the earlier analyses of dendritic growth due to Flemings, Hunt and their co-workers. The most important finding to date is that both the ad hoc optimization principle of minimum undercooling as well as marginal stability considerations yield predictions of the dendrite tip radius which are remarkably close (within 30% of each other with marginal stability considerations yielding the higher tip radius) and in agreement with available experimental data in the literature. More critical experiments are, therefore, needed to distinguish between the predictions of various dendrite characteristics obtained

from these two hypotheses and also between the predictions of the various dendrite growth models proposed in the literature.

Such a test requires simultaneous measurements of both tip radius and tip temperature or the tip radius and tip composition in a single experiment. (Accurate measurements of the tip radius along, or tip temperature or tip composition, separately, cannot be used to distinguish between the various hypotheses/models) Ground-based experiments, employing a model binary Al-Cu alloy, are now being planned in support of this work. This will be the subject of the doctoral thesis of Mr. Li Wang.

Such an experiment has also been proposed (in collaboration with Dr. S.N. Tewari) in response to a flight opportunity announced recently (D-2 mission employing the German Gradient Furnace and Quenching [GFQ] facility). The proposal is still tentative subject to negotiations between NASA and the German Space Agency. Flight experiments are not being planned at this point.

A high temperature directional solidification experimental apparatus is also being designed and built (in collaboration with Dr. S.N. Tewari) in support of this program. A transparent directional solidification apparatus is also being built for direct in-situ observations of dendrite or cell characteristics (in collaboration with Dr. A.M. Chopra).

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The Mathematical and Physical Modelling of  
Electromagnetically Drive Fluid Flow and Associated  
Transport Phenomena in Contained and in Containerless Melts

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Dr. Ernesto Gutierrez  
NAG3-594 (NASA Contact: Fred Harf, LeRC)

The objective of this research is to develop an improved fundamental understanding of electromagnetic, heat and fluid flow phenomena in levitation melted specimens under both earthbound and microgravity conditions. This work is part of the ground based preparatory effort in support of a planned shuttle experiment in 1987.

The research involves the computation of electromagnetic force fields and fluid flow fields in conducting media, resulting from both the passage of a current between two electrodes and induction coils surrounding the melt. This computational work is complemented with experiments using woods metal and tin. Furthermore, the theoretical predictions are also compared with measurements conducted by others.

Important milestones include the following:

- the computation of the electromagnetic force field and fluid flow field in levitation melted specimens both under normal and microgravity
- the computation of the electromagnetic force field and the fluid flow field in inductively stirred melts and a comparison of these data with measurements
- the calculation of the magnetic damping of convection in melts
- the pioneering of the use of hot film anemometry in molten woods metal
- experimental measurements and theoretical predictions concerning the electromagnetic force field and the velocity field in a melt produced by the passage of a current between two electrodes



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### **3. FLUID DYNAMICS AND TRANSPORT PHENOMENA**

Production of Large-Particle-Size Monodisperse Latexes in Microgravity

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NAS8-32951 (NASA Contact: V. Yost, MSFC)  
February 1978 - March 1986

The purpose of this project is to produce large-particle-size monodisperse polystyrene latexes in microgravity in sizes larger and more uniform than can be manufactured on Earth.

A latex is a suspension of very tiny (micrometer-size) plastic spheres in water, stabilized by emulsifiers. The objective of this experiment is to grow billions of these tiny plastic spheres, or balls, to sizes larger than can be grown on Earth, while keeping all of them exactly the same size and perfectly spherical. Thus far on several of the Monodisperse Latex Reactor (MLR) flights, the latex spheres have been returned to Earth with standard deviations of better than 1.4%. The little balls making up these latexes can only be grown in quantity on Earth up to about 5 micrometers in diameter while staying monodisperse because of buoyancy and sedimentation effects. They cannot be stirred sufficiently to keep them in suspension during polymerization because stirring causes shear-induced coagulation which destroys the latex. But in microgravity the absence of buoyancy effects has allowed growth of the balls up to 30 micrometers in diameter thus far during the last flight on STS-11. The MLR has now flown 5 times on the Shuttle, during Missions STS-3, 4, 6, 7, and 11, and three more flights are presently scheduled, to be completed in 1986.

This experiment, the MLR, has now produced the first commercial space product; that is the first commercial material ever manufactured in space and marketed on Earth. The 10 micrometer latex manufactured on STS-6 was officially accepted by the U.S. National Bureau of Standards in July 1984, and offered for sale as the United States National 10 micrometer (10  $\mu$ m) Standard Reference Material. The 30  $\mu$ m-size manufactured on STS-11 was also accepted by NBS and will be offered shortly for sale as Standard Reference Material. NBS has also officially requested that NASA produce for them 30 grams of 30 micrometer latex and 80 grams of 100 micrometer latex which they plan to put on the market. It is hoped that this can be accomplished by the end of the next three flights, that is by 1986.

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## **4. BIOTECHNOLOGY**

## Isoelectric Focusing of Proteins and Peptides

University of Arizona

Dr. Milan Bier

NSG-7333

April 1, 1978 - March 31, 1988

The objective of this research is the development of isoelectric focusing as a process for the purification of proteins and peptides. Isoelectric focusing is a variant of electrophoresis, characterized by exceptionally high resolution.

Electrophoresis has been identified early in the NASA space program as being a process which may benefit from operation in microgravity. As part of this overall project, this laboratory has been committed to the study of the theory and practice of electrophoresis, particularly but not exclusively, as it applies to preparative isoelectric focusing.

The theoretical approach consisted of the development of a unified mathematical model describing in details the electrophoretic mass transport. The model has been reduced to a computer program which predicts the behavior of such systems under a variety of conditions.

The experimental part of the program resulted in the development of three different instruments for preparative isoelectric focusing. In each of these, a different mechanism is utilized for suppression of problems arising from gravity-induced convection. These achievements notwithstanding, isoelectric focusing has not yet been accepted as an alternative to chromatography for preparative purifications.

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## Cell Partition in Two Polymer Aqueous Phases

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NAS8-35333 (NASA Contact: V. Yost, MSFC)  
May 5, 1983 - May 4, 1986

On Earth the sensitive liquid-liquid separation method for biologicals known as aqueous polymer two-phase partition is reduced in efficiency (observed separation/theoretical separation) because gravity induced fluid shear detaches the biologicals from their respective phases during the partitioning process. In low-g buoyancy-driven convection is greatly reduced. Hence, biological separations approaching the theoretical limits should be obtainable. In addition investigations into the factors controlling the low-g demixing and final disposition of the phases will yield information of value to scientists interested in low-g materials processing.

When aqueous solutions of two different polymers are mixed above certain concentrations they frequently form immiscible, liquid, two-phase solutions. Each of these phases usually consists of more than 90 percent water and can be buffered and made isotonic by the addition of low molecular weight species. If a cell or particle suspension is added to such a system in 1-g, then shaken, the cells - upon re-equilibration - are usually found to have partitioned unequally between one of the phases and the interface. Emulsion demixing occurs rapidly due to density differences between the phases. This preferential partition behavior can be used as the basis of a separation procedure for differing cell populations since partition in these systems is determined directly by cell membrane properties.

Manipulating phase system composition separations on the basis of a variety of molecular and surface properties have been achieved, including membrane hydrophobic properties, cell surface charge and membrane antigenicity. Recent work on the mechanism of cell partition has shown there is a randomizing, non-thermal energy present which reduces separation resolution. This stochastic energy is probably associated with hydrodynamic (shear) interactions present during separation.

Studies conducted on board KC-135 aircraft during parabolic maneuvers and by Senator E. J. Garn on board Shuttle flight STS-51D have indicated that in low-g aqueous polymer two-phase emulsions demix by a slow "coalescence" process. Very low fluid shear is present suggesting that low-g partition may be able to resolve cell subpopulations unobtainable, by any method, on Earth.



In low-g two phase emulsions demix to yield one phase floating "yolk-like" completely surrounded by the phase which preferentially "wets" the container wall. Future and ongoing research is aimed at controlling the rate of demixing and final disposition of the phases via both passive means (e.g., altered chamber geometry or polymeric wall coatings with different wetting properties) and active means (electrophoresis of the phases whose interfaces exhibit zeta potentials). In addition variables such as interfacial tension, phase volume ratio and phase viscosity, are being studied to better understand their influence on demixing of the phases in both low-g and 1-g. Polymer two-phase partition results are of value to scientists interested in low-g materials processing of substances such as alloys.

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## Protein Crystal Growth in a Microgravity Environment

University of Alabama, Birmingham

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NAS8-36611 (NASA Contact: John Price, MSFC)

July 1, 1985 - May 31, 1988

The long range objective for this research task is to develop systematic and reliable techniques and hardware for growing protein crystals in space. Studies will be performed to evaluate the potential for enhanced protein crystal growth under microgravity conditions. Fundamental studies of protein crystal growth, both on the ground and in space will be performed in order to identify the major parameters that affect protein crystal growth.

This research program involves a multidisciplinary effort to produce protein crystals in space of sufficient quality and size to permit molecular structural characterization by X-ray crystallography, while simultaneously providing basic ground-based experimental and theoretical supporting research to develop a better understanding of protein crystal growth and to determine if gravity plays a limiting role in the growth process. Beginning with the Apollo program and extending into the Spacelab program, it has been demonstrated that the microgravity environment can provide stable growth conditions than can result in crystals with improved homogeneity and fewer defects. In this program, a variety of proteins will be crystallized on space shuttle flights over a three-year period. Optimum techniques for reliably growing protein crystals under microgravity conditions will be developed. Initially, emphasis will be placed on vapor-diffusion and dialysis techniques, since they can be used with microliter quantities of material, and are two of the most widely used techniques for ground-based growth of protein crystals. Long-range plans include development of new methods for growing protein crystals, based upon the experimental and theoretical studies performed as part of this research program.

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## Cell Partition in Two Polymer Aqueous Phases

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NAS8-35362 (NASA Contact: V. Yost, MSFC)  
August 1, 1983 - July 31, 1986

Partition of biological cells in two phase aqueous polymer systems is recognized as a powerful separation technique which is limited by gravity. A major need of the program is to greatly refine the sensitivity of the technique by developing new polymers that will exhibit a high, selective affinity for specific cell types and which will take advantage of the unique environment of space. Our interest is to synthesize new, selective polymer-ligand conjugates to be used in affinity partition separations.

The two most commonly used polymers in two phase partitioning are dextran and polyethylene glycol. We have performed a thorough review of the chemistry of these polymers, particularly in the area of protein attachment. Preliminary studies indicate the importance in affinity partitioning of minimizing gravity-induced randomizing forces in the phase separation process. The PEG-protein conjugates we are preparing have been demonstrated to be ideally suited for achieving high quality purifications in a microgravity environment. An interesting spin-off of this synthetic work has been the observation of elimination of electroosmosis for PEG-coated capillaries.

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## New Instrumentation for Phase Partitioning

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NAS8-35334 (NASA Contact: V. Yost, MSFC)  
July 1, 1983 - June 30, 1986

Cells and molecules can be purified by partitioning between the two immiscible liquid phases formed by aqueous solutions of polyethylene glycol and dextran. Such purification can be more selective, higher yielding, and less destructive to sensitive biological materials than other available techniques. Earth's gravitational field is a hindering factor as it causes sedimentation of particles to be purified and shear-induced particle randomization. The present proposal is directed toward developing new instrumentation for performing phase partitioning both on Earth and in microgravity.

A major concern of this effort has been a thorough testing of the Ito countercurrent chromatograph obtained from the National Institutes of Health. Examination of the Treffry countercurrent distribution device for automated earth-bound separations has begun. Work has also continued on developing apparatus for microgravity experiments, and one device was flown on STS 51D. A prime concern of this work is to control the rate of phase separation by manipulating interactions between the polymers and coated container walls; coating methods are also under development. This apparatus will be used to take advantage of the new affinity polymers developed in another contract.

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## Purification of Bioactive Pituitary Growth Hormone Cells and Pituitary Growth Hormone Molecules

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NAS9-17416 (NASA Contact: D. Morrison, JSC)

December 1, 1984 - December 1, 1987

The objectives of this research task are: (1) to examine the biological (B) and immunological (I) activities of GH released from electrophoretically purified subpopulations of rat pituitary cells. The effect of microgravity on GH release, as well as on B/I ratio of this secreted hormone will also be studied; and (2) to examine the B/I activities of GH variant forms contained within different subcellular compartments of rat and human postmortem pituitary tissue. The CFES will be used to separate subcellular particles.

The accomplish the first objective, fresh pituitary cells will be prepared from rat pituitary glands by trypsinization. The GH producing subpopulations will be separated by continuous flow electrophoresis (CFE) on the McDonnell Douglas instrument. Separations will be done both on the ground and on Shuttle middeck to confirm previous results of the STS-8 experiment and evaluate GH secretions (and their molecular forms) after exposure to microgravity. The biologic (B) and immunologic (I) activity ratio of the released hormone will be determined by EIA, 3T3 cell adipogenic, and rat tibial line assays. Finally, the purity of the cell fractions will be determined by flow cytometric immunofluorescence procedures.

To accomplish objective 2, human post mortem pituitary glands will be used as the source of tissue for the purification of bioactive bGH. We propose to isolate a hormone form which is low in immunoactivity, but high in biological activity. Purification procedures include HPLC and CFE. In some trials, CFE separations will be done in microgravity where protein loads, and therefore throughput, can be maximized.

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## Cell Maintenance Systems and Inflight Biological Sample Handling

NASA Johnson Space Center  
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In-Center  
April 1984 - continuing task

Mammalian cells are difficult to maintain alive outside of an incubator for more than a few hours. Cells require precise control of temperature, pH, dissolved oxygen, and pressure in their immediate environment. These requirements are modified when cells are taken into microgravity. Normal, non-transformed human cells also require attachment to a suitable surface in order to grow and multiply. Previous experiments on Skylab and STS-8 have tested prototype cell culture and harvesting apparatus.

The Cell Transport Assembly (CTA) flown on STS-8 has required improvements to be able to maintain live cells longer than 48 hours. Methods and small scale apparatus are being developed for harvesting cells and concentrating the resultant cell suspension. Sterile containers, transfer apparatus and procedures are being developed in conjunction with an advanced CTA which can be used to supply living cells for electrophoresis and cell biology experiments.

Automated systems such as the Biological Specimen Test Apparatus (BSTA) are being converted to permit maintenance cultures of both plant and animal cells in the Shuttle middeck lockers. The Refrigerator Incubator Storage Module (RISM) has been modified to hold constant temperatures between 4° and 37°C ± 1 C.

Sterilization systems are being evaluated for use at launch site laboratories and for on-pad operations in the cabin of the Space Shuttle. A portable Ethylene Oxide Generator (EOG) has been modified for sterilization tests with cell culture systems and the Space Bioreactor. Plans include developing other procedures for safe sterilization of the Continuous Flow Electrophoresis System (CFES). New "Steri-loc" sample ports are being designed and developed to allow inflight-sampling from cell culture systems without back contamination. Electric field sterilization methods are being evaluated for use on the Space Bioreactor and other biological holding systems.

## Biological Separations in Space

Johnson Space Center  
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January 1983 - continuing task

NASA has demonstrated the potential of microgravity for the improved processing of useful products. Among such products are biologicals with pharmaceutical value. The Bioprocessing program at JSC has utilized the McDonnell-Douglas Continuous Flow Electrophoresis System (CFES) to separate mixtures of human kidney cells into about 30 fractions. Several of these fractions produce much larger quantities of plasminogen activators than the other fractions. Plasminogen activators are useful in treating blood clot-produced diseases. The goal of the program is to demonstrate that useful cells from a variety of heterogeneous mixtures can be better separated and cultured in space under conditions which allow their biological products to be collected and purified. To achieve this goal, methods and equipment for separation culturing and handling cell cultures in microgravity are being explored. Separation methods include: Continuous Flow Electrophoresis, recirculating isoelectric focusing, and other free-fluid methods which are affected by gravity driven phenomena.

A Continuous Flow Electrophoresis System (CFES) uses a flowing, thin layer of a carrier solution to sweep along a continuous stream of a protein electric field imposed transversely across the layer and are separately collected at the upper end of the chamber. By keeping the layer very thin and by providing precise cooling, convective mixing is minimized. Even so, for effective processing in normal gravity, the density of the sample stream and of the carrier fluid must be nearly identical. Since the density of most proteins is greater than that of the carrier field, if the concentration of the protein in the sample is excessive, gravity causes the sample to collapse around the inlet part of the device. Therefore on Earth, the sample must be diluted to less than 0.2% protein. In space, the sample can be concentrated to 25% protein. By merely increasing the protein concentration, 125 times more material can be separated in null gravity per unit time than in normal gravity.

In addition, in space, gravity-driven convection currents cease to operate and the thickness of the layer and the size of the sample inlet can be increased from approximately 0.5 mm to 1 mm. By doubling the diameter of the inlet, four times as much cross-sectional area is gained. Therefore, with the 125 times greater concentration, about 500 times more material can be

separated per unit time in null gravity than in normal gravity. Moreover, higher voltages (which produce more heating) and longer residency times (slower carrier fluid flow) can be employed in space because of the lack of buoyancy-driven mixing.

The advantage of null gravity for the CFES separation of proteins are even greater in the separation of cells. Johnson Space Center, under a Joint Endeavor Agreement with McDonnell-Douglas, used the CFES during the STS-8 mission to separate human embryonic kidney cells.

The embryonic kidney is composed of a heterogeneous mixture of cells with different functions. Different cells produce different specific biomolecules (e.g., hormones, enzymes, and other chemical messengers). The goal of the cell separation experiment was to isolate cells that produce a high quantity of a desired substance and then to culture the isolated cells and obtain the substance from the growth medium. The main substance of interest is urokinase, an enzyme that is used to dissolve blood clots. However, JSC is also examining the various fractions for other substances produced in the kidney (e.g., erythropoietin, tissue activator, nonspecific viral inhibitors).

In addition, pituitary cell samples were separated on STS-8 for the purpose of obtaining quantities of growth-hormone-producing cells sufficient for biological assays. Radioimmunoassay, the standard assay for growth hormone, fails to detect more than 90% of the biologically active forms of the hormone. The space separation experiments will help investigators determine forms of the hormone that are active in the body.

Other experiments now being developed for space include separation and culture of hybridoma cells producing monoclonal antibodies and various subsets of lymphocytes involved in transplant rejection.

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## Kidney Cell Electrophoresis in Microgravity

Pennsylvania State University  
Bioprocessing and Pharmaceutical Research Center  
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NAS9-17431 (NASA Contact: D.R. Morrison, JSC)  
May 1, 1985 - May 1, 1988

The objectives of this research task are: (1) provide ground-based cell electrophoresis technology and cell culture support for electrophoretic purifications of cultured human embryonic kidney cells in microgravity; (2) develop flow cytometric methods for analyzing and sorting of human embryonic kidney cells; and (3) perform biophysical analyses of purified cultured human embryonic kidney cells returned from space flight.

To accomplish the first objective, cultured human embryonic kidney cells, in early passage will be subjected to continuous flow electrophoresis. The effects of content of divalent cations, antibiotics, and neutral additives will be studied in low conductivity electrophoretic separation experiments. Cells from various lots will be examined and compared with respect to their ability to multiply, to produce plasminogen activators, to differentiate morphologically, and to retain differentiated function in vitro.

To accomplish objective two, a laser flow cytometer will be used to quantify cells that produce plasminogen activator on a one-by-one basis by staining them with fluorogenic amide substrates that release 4-methoxy-2-naphthylamine which can be precipitated intracellularly by the addition of 5-nitrosalicylaldehyde. The percent plasminogen activator producing cells will be quantified in early-passage cultures and purified cell suspensions by this method. The light scattering signature in flow cytometry of electrophoretically purified cell subpopulations will be determined in order to establish whether or not there exist basic morphological criteria that may also be used as a means of cell purification by viable cell sorting. A flow cytometer will also be used to examine the relationship between cell surface charge and cell function by staining simultaneously with fluorescent poly-L-lysine and plasminogen activator fluorogenic substrates.

To accomplish objective three, post-flight biophysical analyses of cells purified in space, the above mentioned methods will be used. The percent cells producing each of the three different types of plasminogen activator will be identified in each fraction using specific markers in flow cytometry. Cell life analysis using DNA staining and flow cytometry of cells from each purified fraction will be used to reveal which fractions retain reproductive potential.

The electrophoretic mobility of cells from each purified fraction will be confirmed on the basis of analytical cell electrophoresis using an automated electrokinetic analyzer.

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Cells from Mammalian Tissues," 1986, submitted.

## **5. GLASSES AND CERAMICS**

## Containerless Processing of Glass Forming Melts in Space

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NAS8-34758 (NASA Contact: V. Fogle, MSFC)  
April 1983 - June 1987

The major objectives of this work are to: (1) obtain quantitative evidence for the suppression of heterogeneous nucleation/crystallization in containerless melts in micro-g; (2) study melt homogenization in the absence of gravity driven convection; (3) develop the procedures for preparing precursor samples suitable for flight experiments; (4) perform comparative property analysis of glasses melted on earth and in micro-g; (5) determine the feasibility of preparing glass shells in micro-g for use as laser fusion targets; and (6) assess the operational performance of the single axis acoustic levitator/furnace apparatus for processing multi-component, glass-forming melts in micro-g.

If the heterogeneous nucleation/crystallization of a melt is suppressed by containerless melting, then its critical cooling rate ( $R_C$ ) for glass formation in micro-g will be less than its  $R_C$  on earth. The practical consequence of a smaller  $R_C$  for glass formation in micro-g is an extension of the compositional limits for glass formation and the possibility of obtaining new glasses by melting in micro-g. For samples returning as glass after containerless melting in micro-g, the ratio of  $R_C$  on earth to the cooling rate ( $R$ ) used in micro-g will serve as a quantitative measure of the degree to which glass formation is enhanced, or heterogeneous nucleation is suppressed. Binary calcia-gallia and lead-silicate and ternary calcia-gallia-silica compositions possessing different critical cooling rates will be heated, melted, and quenched in an acoustic levitator/furnace for the MEA/A-3 experiment. A wide range of physical, optical, thermal, and mechanical properties will be measured for glasses made in micro-g for comparison with the same properties of glasses made on earth. A borosilicate glass sphere containing an irregularly shaped air bubble will be remelted in micro-g in order to examine the feasibility of producing glass shells of thin uniform wall thickness. Melt homogenization in the convection-free environment of micro-g will be investigated by observing the level of chemical homogeneity achieved in melts made from deliberately inhomogeneous precursor samples.

An important practical task is to determine the suitability of using hot pressed precursor samples for containerless melting experiments in micro-g. Hot pressing has the advantage of being a relatively simple way of preparing precursor samples without chemical contamination

from a container. The degree of chemical inhomogeneity that can be tolerated in a hot pressed precursor while still yielding a chemically homogeneous multi-component melt within a reasonable time in micro-g is being determined.

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Ray, C. S., Day, D. E., and Chakraborty, I. N., "Crystallization of  $La_2O_3$ - $B_2O_3$  Glasses Containing  $Al_2O_3$  or  $Y_2O_3$ ," presented at XIV International Congress on Glass, New Delhi, March 1986.

Crystallization, Optical, and Chemical Properties of Fluoride Glasses

Rensselaer Polytechnic Institute  
Dr. Robert H. Doremus  
JPL-955870 (NASA Contact: T.G. Wang, JPL)

The objectives of this program are to: (1) cooperate with JPL in constructing an ACES apparatus and to prepare samples and experiments for space flight; (2) measure properties needed for the flight; and (3) examine the nucleation and crystallization kinetics and other properties of fluoride glasses on the ground, so these properties can be understood and compared with containerless behavior.

Fluoride glasses have great promise as infrared optical components, especially fibers, because they are transparent to 8  $\mu$ m and higher. In order to optimize properties, different glass compositions are needed. Some are hard to form in a container, and may possibly be formable in a containerless furnace. Understanding of crystallization with and without a container could lead to glasses with optimum properties. Chemical durability (attack by water) can limit or extend the applicability of fluoride glasses.

Progress to date includes: (1) melting and casting of fluoride glasses for ground studies and for flight STS-11; (2) measurement of crystallization kinetics with the DSC; (3) examination of microstructure of crystallized sample; (4) cooperation with JPL in building the ACES apparatus; (5) post flight analysis of video tape, temperature, and sample microstructures from ACES experiment on STS-11. A report has been prepared; and (6) work is underway on samples and equipment for the next flight.

Publications

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## Physical Phenomena in Containerless Glass Processing

Clarkson University  
Dr. R. S. Subramanian  
Dr. Robert Cole  
NAS8-32944 (NASA Contact: V. Fogle, MSFC)  
December 1977 - September 1986

The objective of this research is to study the behavior of gas bubbles inside drops of model fluids and molten glasses in free fall, focusing on their migration and interaction. Such migration will be induced by thermocapillary rotation and/or oscillation of the drop, and other means. The results of the experiment are expected to be of use in the development of techniques for mixing and fining glasses in space and in providing a better understanding of how microballoons are formed.

Experiments on bubbles contained within drops are scheduled for flight aboard the Shuttle in late December 1985. In these experiments, to be performed in the 3-Axis Acoustic Levitator (3-AAL) designed and built at the Jet Propulsion Laboratory, drops of model fluids containing gas bubbles will be rotated or spot-heated. The resulting activity within the drop will be recorded on videotape for later analysis on ground.

Ground-based research has been conducted on the various physical phenomena of relevance to the space experiments. The migration of drops and bubbles caused by capillarity as well as that induced by rotation has been modelled. Experiments have been conducted on thermocapillary motion in model fluids and glass melts, and on bubble and drop behavior in rotating liquids. Other experiments on gas bubble migration in a thermal gradient and bubble behavior in a settling drop are in progress.

### Publications

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## 6. COMBUSTION SCIENCES

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Combustion of Particulate Clouds at Reduced Gravitational Conditions

University of California, San Diego  
Dr. A. L. Berlad  
NAG3-473 (NASA Contact: Howard Ross, LeRC)

The combustion of clouds of particulates represents an area of combustion of fundamental and practical importance. Microgravity experimental conditions are required in order to create suitably uniform clouds of selected particulates and then to study flame propagation and extinction associated with these uniform clouds. This work is needed in order to characterize experimentally the fundamental combustion properties of important particulates, to test central theoretical constructs associated with flame propagation and extinction theory, and to understand the effects of gravitationally-induced processes on particulate combustion phenomena.

Ground-based and experimental and analytical efforts are required to develop and implement experimental apparatus, methods of study and the analytical tools required to successfully carry out a Space Shuttle-based study of combustion of clouds of particulates at reduced gravitational conditions.

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## Scientific Support for a Space Shuttle Droplet Burning Experiment

Princeton University  
Professor F. A. Williams  
Professor F. L. Dryer  
NAS3-24640 (NASA Contact: Kurt Sacksteder, LeRC)  
June 20, 1985 - November 30, 1986

The general objective of this program is to ascertain how best to make use of reduced gravity to pursue scientific investigations of droplet combustion. The specific objective is to provide scientific support during development of a droplet burning experiment that is to be carried out in the Space Shuttle. The planned experiment is intended to improve our understanding of droplet combustion, especially in relationship to time-dependent and extinction phenomena.

The research tasks include theoretical modeling of droplet burning, ground-based experimentation on droplet burning, support to NASA in providing advice on hardware aspects of the flight experiment and analysis of data to be obtained in the experiment. The modeling addresses questions related to burning rates and to ignition and extinction phenomena. The ground-based experiments are focused on droplet ignition and on impulses imparted to droplets by ignition sparks; spark designs for minimum impulse are addressed. The support activities include advisory participation in planning and in implementation of the flight experiment on droplet combustion.

### Publications

Williams, F. A., "Studies of Experiments on Droplet Burning at Reduced Gravity," in Proceedings of 4th European Symposium on Material Sciences under Microgravity, Madrid, 1983, ESA SP-191, pp. 191-196.

Peters, N. and Williams, F. A., "Effects of Chemical Equilibrium on the Structure and Extinction of Laminar Diffusion Flames," in Dynamics of Flames and Reactive Systems; Progress in Astronautics and Aeronautics 95, AIAA, 1984, pp. 37-60.

Williams, F. A., "Ignition and Burning of Single Liquid Droplets," Acta Astron. 12, 547 (1985).

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**C. PHYSICS AND CHEMISTRY EXPERIMENTS (PACE)**

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Scientific Support for an Orbiter Middeck Experiment on  
Solid Surface Combustion

University of Kentucky  
Professor Robert A. Altenkirch  
Dr. M. Vedha-Nayagam  
NAS3-23901 (NASA Contact: K. Sacksteder, LeRC)  
December 24, 1984 - April 3, 1988

The overall objectives of the experiment are to: (1) determine the mechanism of gas-phase flame spread over solid fuel surfaces in the absence of any buoyancy induced or externally imposed gas-phase flow; and (2) improve the fire safety aspects of space travel.

The spread of flame in the gas over the surface of a solid combustible involves in an essential way the transfer of heat from the flame to the solid fuel immediately ahead of it. This heat transfer is affected by the character of the gas-phase flow, and so the phenomenon of flame spreading under reduced gravity, in which the flow is generated by gasification of the solid combustible, is apt to be different from what occurs under the Earth's normal gravitational acceleration where the flow is largely buoyancy driven.

An experiment has been designed for the Middeck of the Space Shuttle to aid in understanding the process of flame spreading in the absence of a buoyancy-driven flow. A chamber approximately  $0.035 \text{ m}^3$  in volume is to contain either a thin sample of a cellulosic material or a thick sample of polymethylmethacrylate and an oxidizing environment of  $\text{O}_2$  and  $\text{N}_2$ . Samples will be ignited at one end, and the ensuing flame spread process will be filmed. The spread rate can be determined from the films, and surface and gas-phase temperatures just above the surface will also be recorded. A matrix of eight experiments to be carried out on the Middeck has been identified. These data will help to clarify the mechanism of forward heat transfer in the low-gravity flames.

The experimental apparatus has been constructed at NASA's Lewis Research Center and tested in the Drop Tower facilities. Current testing is being carried out at the University of Kentucky. Methods of data reduction are being developed as are theoretical analyses of reduced-gravity flame spread problem.

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## Publications

Vedha-Nayagam, M. and Altenkirch, R. A., "Gravitational Effects of Flames Spreading Over Thick Solid Surfaces," Acta Astron. 12, 565-572 (1985).

Vedha-Nayagam, M. and Altenkirch, R. A., "Backward Boundary Layers in Downward Flame Spread," in Proceedings of 20th International Symposium on Combustion, Pittsburgh, 1985, pp. 1583-1590.

## Presentations

Altenkirch, R. A., "Combustion Studies in Microgravity," presented at National Science Foundation Workshop on Opportunities for Academic Research in a Low-Gravity Environment, Washington, DC, July 1985.

Altenkirch, R. A., "Flame Spread under Microgravity Conditions," presented at Gordon Research Conference on Gravitational Effects in Materials and Separation Processes and Living Systems, New London, NH, August 1985.

Altenkirch, R. A., Short, J. L., Vedha-Nayagam, M., and Padgaonkar, A. M., "Including Radiation Heat Transfer Effects in Dimensionless Flame Spread Correlations," presented at Central States-Western States Sections/The Combustion Institute Joint Technical Meeting, San Antonio, April 1985.



## Free Surface Phenomena Under Low- and Zero-Gravity Conditions

University of California, Berkeley  
Professor Paul Concus  
Professor D. Coles, Cal Tech  
Professor R. Finn, Stanford University  
Professor L. Hesselink, Stanford University  
NAG3-147 (NASA Contact: Dr. A.T. Chai, LeRC)  
February 28, 1985 - January 31, 1986

In a low- or zero-gravity environment the free surface of a liquid can behave in striking, unexpected ways. For example, in a partially filled container, a free surface that is well behaved under terrestrial conditions can rise to an arbitrarily large height or even fail to exist when gravity is absent. This study attempts to answer the central mathematical questions: Under what conditions can free surfaces exist and what are their properties? -- and experimental questions of what means can be devised to observe and to measure the surfaces quantitatively.

Current activity focuses on material selection and the design of optical diagnostic methods for in-space experiments. These experiments will test mathematical predictions of discontinuous transition from existence to non-existence of capillary free surfaces in certain geometric configurations under zero gravity conditions. Liquids and container materials suitable to achieving the required contact-angle range and optical properties are being investigated and, as well, the effects of contaminants and fluid motion on contact angle. The optical diagnostic technique investigation concerns laser-induced fluorescence, including emphasis on data acquisition, sources and detectors, reliability, data management, and computer control.

### Publications

Concus, P. and Finn, R., "On the Extremals of a Subsidiary Capillary Problem," J. Reine Angew. Math. 353, 215-220 (1984).

Finn, R., "A Subsidiary Variational Problem and Existence Criteria for Capillary Surfaces," J. Reine Angew. Math 353, 196-214 (1984).

Mass Transport Phenomena Between Bubbles and Dissolved Gases  
in Liquids Under Reduced Gravity Conditions

University of Toledo

Professor Kenneth J. De Witt

J. L. Brockwell, Union Carbide Technical Center

NAG3-34 (NASA Contact: Dr. A.T. Chai, LeRC)

November 15, 1983 - January 14, 1987

The long term objective of the experiment is to observe the dissolution of isolated immobile bubbles of specified size and composition in a solvent liquid of known concentration in the reduced gravity environment of earth orbit. Preliminary bubble dissolution experiments conducted both in the NASA Lewis 2.2 sec. drop-tower and in normal gravity using the SO<sub>2</sub>-Toluene system were not completely successful in their objective. The method of gas injection and the lack of bubble interface stability experienced due to the extreme solubility of SO<sub>2</sub> in Toluene had the effects of changing the problem from that of bubble dissolution to one of bubble formation stability and subsequent dissolution in a liquid of unknown initial solute concentration.

Current work involves further experimentation in order to refine the bubble injection system and to verify the concept of having a bubble with a critical radius in a state of unstable equilibrium. The method of bubble injection is continuing to be that of syringe injection, which is acceptable at this stage of the feasibility study. The critical radius concept is of major importance since it is needed for initialization for all experiments involving highly soluble gas-liquid systems. In these systems, the high gas solubility generally prevents the formation of a stable gas-liquid interface, so that a bubble can be formed, until a suitable local background concentration of the dissolved gas in the liquid has been attained. This background concentration is not uniform throughout the liquid, which makes subsequent bubble dissolution data of less value than desired. The critical radius concept is ready to be tested using the CO<sub>2</sub>-Toluene system in normal gravity. An improved prototype experiment package has been designed and constructed for this purpose at NASA Lewis. Bubble rise will be prevented by the use of very fine fibers. After establishment of a bubble in critical equilibrium is achieved in normal gravity, Lear Jet tests (23 sec. of free-fall time) will be conducted in order to refine the injection system and to determine whether a critical bubble can be stabilized in this time period. Finally, injection hardware will be further examined in the NASA Lewis drop-towers, and work will begin in the conceptual design of the middeck experiment.

The total experiment will involve the injection of a single bubble of gas of approximately a prescribed size and composition into a quantity of thermostatted liquid under controlled pressure conditions. The pressure on the liquid is then adjusted to maintain the bubble in a state of unstable equilibrium with the surrounding liquid. As a result of a step increase in the pressure, bubble dissolution is initiated. The rate of mass transfer can be determined from an observation of the change in bubble size with time.

Determination of the Correlation Length in Helium II in a Microgravity Environment

University of Oregon  
Dr. Russell J. Donnelly  
Dr. Charles E. Swanson  
(NASA Contact: D. D. Elleman, JPL)  
July 1, 1985 - June 30, 1986

The objective of this research is to assess a method of measuring the circulation length of helium II in space.

The superfluid properties of liquid helium are associated with the properties of a complex order parameter  $\psi$ , which has amplitude and phase. The distances over which the amplitude and phase are strongly correlated are referred to as correlation or coherence lengths  $\xi$  and these lengths are thought to diverge as the temperature  $T_\lambda$  approaches the lambda transition  $T_\lambda$  of liquid helium. The coherence length is fundamental to many properties of liquid helium: (1) it enters the renormalization group calculations of the specific heat of liquid helium; and (2) it determines the scale needed to build He II Josephson junctions, the scale of fluctuations near the lambda transition (which are studied by light scattering), the size of quantized vortex cores, the distance over which superfluidity vanishes near a wall, and the scale of "size effects" which shift the lambda transition in confined geometries.

At the present time the coherence length is known in order of magnitude, but not with precision. The coherence length coefficient  $\xi_0$  is seen to vary by a factor of  $\sim 4$  among different experiments. One reason is the influence of gravity which sets a limit on how close to  $T_\lambda$  one can carry out a precise experiment in a container of given height. Indeed much of the same technology to be used for the specific heat experiment in space is directly useful in coherence length measurement for the JPL space cryostat and the Lipa subnanodegree thermometer.

Tasks will include bibliographic research, consulting with experts in the field, numerical simulation of the experiment, and preliminary experiments.

Publications

Homsy, G. M. and Donnelly, R. J., "Fluid Mechanics and Fluid Physics in a Low-Gravity Environment," in Opportunities for Academic Research in a Low Gravity Environment (M. Summerfield, ed.), AIAA, 1986 (in press).

## Buoyancy Effects Upon Vapor Flame and Explosion Processes

Science Applications International Corporation  
Dr. Raymond B. Edelman  
Dr. M. Yousef Bahadori  
NAS3-22882 (NASA Contact: Sandra Olson, LeRC)  
November 1984 - February 1986

The overall objective of this microgravity project is to develop experimental data and theoretical analyses critical to the understanding of the effects of buoyancy on fuel-air mixing, flame intensity and flame propagation in laminar and turbulent gas jet diffusion flames. Collateral objectives include developing information relevant to the control of unconfined fires in outer space and on earth as well as providing information needed to support the development of advanced low momentum burners and combustion chambers for industrial applications.

Current research has involved the preparation of the Science Requirements Document in which prior research conducted on this project has been integrated to provide a set of objectives including experimental and theoretical elements to be achieved in conjunction with an experiment to be conducted on the Space Shuttle. In addition, the conceptual design of the experiment is being prepared. This effort is resulting in the preparation of the Conceptual Design Document which, in addition to addressing the chamber, fuel management system, ignitor, instrumentation and data storage aspects of the experiment, is also addressing shuttle requirements, astronaut interfaces, safety, and post-flight data analysis.

### Publications

Edelman, R. B. and Bahadori, M. Y., "Effects of Buoyancy on Gas Jet Diffusion Flames - Experiment and Theory," presented at 36th IAF Congress, Stockholm, Sweden, October 1985, IAF 85-288.

## Cryogenic Equivalence Principle Experiment

W. W. Hansen Laboratories of Physics  
Dr. C.W.F. Everitt  
Dr. Paul W. Worden

The objective of this research is to test the equivalence of inertial and passive gravitational mass in an earth-orbiting satellite. Preliminary work and technology development is being done in a ground-based experiment which is expected to test the equivalence principle to a few parts in  $10^{13}$ ; a satellite version might have a sensitivity of one part in  $10^{17}$ .

The ground-based experiment is now well developed. It consists of comparing the motions of two cylindrical test masses suspended in precision superconducting magnetic bearings and free to move along the horizontal (axial) direction. The masses are made of niobium and lead-plated aluminum. A position detector based on a SQUID magnetometer measures the differential motion between the masses. The periods of the masses are matched by adjustment of the position detector until the system is insensitive to common mode signals, and so that the experiment is less sensitive to seismic vibration. The apparatus is contained in a twelve inch helium dewar suspended in a vibration isolation stand. The stand achieves 30 db isolation from horizontal motions between 0.1 and 60 Hz, by simulating the motion of a 200 meter long pendulum with an air bearing. With this attenuation of seismic noise and a common mode rejection ratio of  $10^5$  in the differential mode, the ground based apparatus should have a sensitivity to equivalence principle violations of one part in  $10^{13}$ . The primary limitation is due to seismic noise.

The earth-based apparatus will be appropriately scaled and modified for operation in zero gravity. The test masses will be about 10 centimeters in diameter. A crucial difference in the orbital experiment is the effect of the gravity gradient of the earth on the masses. This can be eliminated by putting the centers of mass of the test bodies at the same location. If the centers of mass are not coincident, the resulting acceleration can be detected and used as a error signal for a servo loop to drive them into coincidence. The Shuttle version of the experiment should have a sensitivity of about  $10^{-15}$  limited by the vibration environment and gravity gradient field of the Shuttle orbiter. An independent drag-free satellite is necessary for the ultimate version of the experiment which might exceed a sensitivity of  $10^{-17}$  limited by gas pressure effects.

## Publications

Worden, P. W., "Almost Exactly Zero: The Equivalence Principle," Near Zero, 1986 (in press).

Light Scattering Tests of Fundamental Theories of Transport Properties in the Critical Region

University of Maryland  
Professor R. W. Gammon  
Dr. M. R. Moldover, NBS  
NAG3-638 (NASA Contact: Dr. R. Lauver, LeRC)  
May 1, 1985 - April 30, 1986

The objective of this research is to measure the decay rates of critical density fluctuations in a simple fluid (xenon) very near its liquid-vapor critical point using laser light scattering and photon correlation spectroscopy. Such experiments have been severely limited on earth by the presence of gravity which causes large density gradients in the sample when the compressibility diverges approaching the critical point. The goal is to measure fluctuation decay rates deep in the critical region, to within 3 microKelvin if the residual acceleration is low enough. The scaled wavevector would be as large as 1000. This will require loading the sample to 0.01% of the critical density and taking data as close as 3 microKelvin to the critical temperature ( $T_c = 289.72$  K). Other technical problems have to be addressed such as multiple scattering and the effect of wetting layers.

We have demonstrated the ability to avoid multiple scattering by using a thin sample (100 microns), and a temperature history which can avoid wetting layers, satisfactory microcomputer controlled temperature control and measurement, and accurate sample loading. Thus the questions of experimental art are solved leaving the important engineering tasks of mounting the experiment to maintain alignment during flight and preventing g-jitter from distorting the sample.

The experiment consists of measurement of the scattering intensity fluctuation decay rate at two angles for each temperature while simultaneously recording the scattering intensities and sample turbidity (from the transmission). The intensity and turbidity data is used to measure the correlation range at each temperature and to locate  $T_c$ . Temperatures will cover from 100 milliKelvin to the resolution of the bridges, 3 microKelvin from the critical point.



Investigation of Surface Driven Convection as a Feasibility Study for a Zero Gravity Experiment

University of Texas at Austin  
Dr. E. L. Koschmieder  
NAG3-393 (NASA Contact: Dr. A.T. Chai, LeRC)  
January 1, 1985 - December 31, 1985

The objective of this research is to establish in the laboratory in the presence of gravity the characteristics of the onset of surface driven convection, and to determine the planform of the convective motions.

A shallow layer of silicone oil of less than 2 mm depth is heated uniformly from below, while its upper surface is cooled uniformly from above through a very thin layer of air. The first experiments were done with a very wide layer in which the consequences of the lateral wall of the fluid were negligible. The current series of experiments is concerned with the onset of convection in narrow circular and square containers, the configuration which ultimately will be used in a zero gravity experiment.

Publications

Koschmieder, E. L. and Biggerstaff, M. I., "Onset of Surface Tension Driven Benard Convection," J. Fluid Mech., 1985 (accepted).

## Lambda Point Experiment

Stanford University  
Dr. John A. Lipa  
Dr. William M. Fairbank  
NAG2-62 and JPL 955057

The objective of the research is to perform a new test of the theory of cooperative or second order, phase transitions by making use of the microgravity conditions on the Shuttle, the JPL SL-2 Helium Dewar, and the Stanford High Resolution Thermometer.

Central to the study of cooperative transitions is the idea of asymptotic behavior of various thermodynamic properties in the limit as the temperature interval from a transition is reduced to zero. Most current theoretical predictions are made in this limit. The present experiment is designed to explore the submicrodegree region of this transition using new thermometry technology which pushes the resolution of temperature close to the fundamental limits set by statistical fluctuations.

In the initial experiment, the plan calls for the observation of the temperature dependence of the heat capacity of helium very near the lambda point with a resolution of a few times  $10^{-10}$  deg. This experiment will be conducted in conjunction with JPL, who will provide the helium dewar. A second dewar will be constructed to allow follow-up experiments with a turn-around time of the order of one year. Additional experiments will be developed to probe other properties of helium, among them the superfluid Josephson effect, tricritical point behavior, and transport properties. At a later phase, the use of a low cost, recoverable spacecraft, such as the Spartan, will make it more desirable to perform experiments in a quieter environment than the Shuttle.

### Publications

Lipa, J. A., "Prospects for High Resolution Measurements Near the Lambda-Transition in Space," in Proceedings of 75th Jubilee Conference on Helium, Chapter 4 (J.G.M. Armitage, ed.), World Scientific, 1984, p. 206.

Critical Transport Properties in Liquid Helium under Low Gravity

Duke University  
Dr. Horst Meyer  
Dr. Robert Behringer  
NAG-379 (NASA Contact: Stephen Castles, GSFC)

The objectives of this research task are: (1) a measurement of the shear viscosity of  $^3\text{He}$  and  $^4\text{He}$  and mixtures near their liquid-vapor critical point, and near the superfluid transition; (2) feasibility study of such measurements for a space shuttle flight; and (3) study of the viscosity unaffected by gravity.

A torsional oscillator operating at a frequency of 150 Hz has been used to measure the shear viscosity along the critical isochore of these fluids under conditions of high temperature stability (approximately 1 K). The liquid-vapor critical points are  $T_c = 3.3$  K and 5.2 K for  $^3\text{He}$  and  $^4\text{He}$ . The stability of the oscillator permits data with a scatter of less than 0.1% to be obtained. Preparation of the correct density is monitored by a density cell in thermal contact with the oscillator. We have completed the measurements on  $^3\text{He}$  and  $^4\text{He}$  near  $T_c$  and are progressing with the comparison with predictions in the presence of the earth's gravity. Preliminary results are very encouraging. The effect of gravity is more pronounced for these two fluids than for any other fluid.

Publications

Agosta, C., Cohen, L. H., and Meyer, H., "The Shear Viscosity of  $^3\text{He}$  and  $^4\text{He}$  Near the Liquid Vapor Critical Point," Bull. Am. Phys. Soc. **30**, 713 (1985).

## Precise Viscosity Measurements Very Close to Critical Points

National Bureau of Standards

Dr. M. R. Moldover

Dr. R. F. Berg

Professor R. W. Gammon, University of Maryland

C-86129D (NASA Contact: Dr. A.T. Chai, LeRC)

We are examining the feasibility of performing precise viscosity measurements very close to the critical point of a pure liquid in the Space Shuttle's Low-gravity environment. To this end, we have developed and successfully operated a torsion oscillator viscometer operating at moderate temperatures (0-100 C) and pressure 0-10 MPa) using a low frequency (0.5 Hz) and shear rate of  $0.05 \text{ s}^{-1}$ . A viscosity resolution of 0.2% is allowed to determine the viscosity exponent of a critical mixture of methanol and cyclohexane to within 2%. Preliminary measurements on the pure fluid system of sulfur hexafluoride pose the challenges of sample stirring and long equilibrium times for mass redistribution near the critical point. The role of gravitational stratification in limiting the temperature resolution has been demonstrated. These effects as well as apparatus concerns such as miniaturization and mechanical stability are now under study.

### Publications

Berg, R. F. and Moldover, M. R., "Viscosity Measurements Near a Critical Point Using a Novel Torsion Oscillator," Int. J. Thermophys. 7, 675 (1986).

Moldover, M. R., "Opportunities for Low-Gravity Experiments in Critical Phenomena," in Proceedings of National Science Foundation Workshop on Opportunities for Academic Research in a Low-Gravity Environment, AIAA, 1986 (in press).

Berg, R. F. and Moldover, M. R., "Viscometer for Low-Frequency Low-Shear Rate Measurements," Rev. Sci. Instrum., 1986 (in press).

## Impurity Extraction by Droplets

National Bureau of Standards  
Dr. Graham Morrison  
Dr. J. M. Kincaid

The objectives of this research task are to model and to measure the phase equilibrium of a finely divided fluid containing a large number of chemically similar species. The objective is to develop an accurate, usable model for such phenomena as pollutant extraction of rain clouds, industrial separation in spray towers, and separation in emulsions. The project has been designed as a hierarchy of complementary theoretical and experimental steps.

A theory has been developed to describe the segregation of complex impurities at the interface of a solvent. This phenomenon is important in phase behavior when a large fraction of molecules in a material are near an interface, the situation in a finely divided material. The theory will be modified to account for the effect of surface curvature on the surface tension. The study of mixtures differs from pure fluids not only because of the surface effects but also because composition differences between the droplet and the surrounding vapor can stabilize a droplet with respect to a bulk phase.

The experiment has been attacked on three fronts. First, we began the construction of a device to make precise measurements on earth of the bulk-phase equilibria of the mixture we proposed to study as a mist. Second, we have begun to work with mists and to design an appropriate apparatus for doing a series of terrestrial experiments on them. Finally, we have developed a theory for interfaces of very complex mixtures.

The bulk apparatus has been completed with the exception of correcting several design flaws. Some preliminary measurements have been made on n-butane for the purpose of calibration; measurements on complex mixtures will be pursued this year. We have begun to work with mists of water in air to learn how to handle such materials. The mist is generated by a piezoelectric device which produces droplets in the range 1 to 10 microns in diameter. While working with the mists, we have encountered several striking phenomena, movement of the droplets through interfaces, the formation of bundles and chains of droplets, and the stability of the water mists in organic liquids.

Finally, we have completed a theory to describe interfacial behavior in very complex mixtures. The model is a perturbation model based on the van der Waals squared-gradient model for pure fluids. In our efforts to model the

carbon dioxide/hydrocarbon system we have found that, given the limitation on the precision of our chemical analysis technique, the upper bound on the mist of droplet size is 0.1 microns--a regime where Brownian motion, not settling in a gravitational field, dominates the behavior of the droplets.

## Surface Tension Driven Convection

Case Western Reserve University  
Professor Simon Ostrach  
Professor Y. Kamotani  
NAG3-570 (NASA Contact: T.P. Jacobson, LeRC)  
August 1984 - July 1986

The objective of the investigation is to design a thermocapillary experiment to study the transient and steady-state flows in the long-duration low-g environment of the Shuttle.

The basic configuration is one in which a fluid contained in a circular dish is subjected to an imposed surface heat flux to generate the thermocapillary flow. Quantitative data will be obtained on the nature and extent of the flows, the effects of heating rates on the flows, the effects of different thermal signatures, the effects of surface deformations, and on flow oscillations. Ground-based and drop-tower experiments together with a numerical analysis have been made to provide base data and to ensure that the operating conditions and the configuration will lead to flows that can reasonably be observed and measured. The configuration to be tested is of further importance because it differs from others in which flow oscillations were observed.

### Publications

Fu, B-I. and Ostrach, S., "Numerical Solutions of Thermocapillary Flows in Floating Zones," in Transport Phenomena in Materials Processing, Volume 10, ASME, 1983.

Kamotani, Y., Ostrach, S., and Vargas, M., "Oscillatory Thermocapillary Convection in a Simulated Floating-Zone Configuration," J. Cryst. Growth **55**, 83-90 (1984).

Balasubramanian, R. and Ostrach, S., "Fluid Motion in the Czochralski Method of Crystal Growth," PCH Physicochem. Hydrodyn. **5**, 3-18 (1984).

Ostrach, S., Kamotani, Y., and Lai, C. L., "Oscillatory Thermocapillary Flows," PCH Physicochem. Hydrodyn. **6**, 585-599 (1985).

Kamotani, Y., Lin, J. K., and Ostrach, S., "Effect of Destabilizing Heating in Gortler Vortices," J. Heat Transf. **107** (1985).

Kamotani, Y., Wang, L. W., Ostrach, S., and Jiang, H. D., "Experimental Study of Natural Convection in Shallow Enclosures with Horizontal Temperature and Concentration Gradients," Int. J. Heat & Mass Transf. 28 (1985).

#### Presentations

Lai, C. L., Ostrach, S., and Kamotani, Y., "The Role of Free-Surface Deformation in Unsteady Thermocapillary Flow," presented at 1985 U.S.-Japan Heat Transfer Joint Seminar, San Diego, September 1985.

Balasubramanian, R. and Ostrach, S., "Transport Phenomenon Near the Interface of a Czochralski Crystal," to be presented at 1986 ASME Winter Annual Meeting, December 1986.

Kamotani, Y. and Kim, J., "effect of Zone Rotation on Oscillatory Thermocapillary Flow in Floating Zone Process," to be presented at 1986 ASME Winter Annual Meeting, December 1986.

Kamotani, Y. and Sahraoui, T., "Oscillatory Natural Convection in Rectangular Enclosures Filled with Mercury," to be presented at Joint ASME/JSME Thermal Engineering Conference, March 1987.



## Studies in Electrohydrodynamics

Princeton University  
Professor Dudley A. Saville  
NAG3-259 (NASA Contact: Dr. A.T. Chai, LeRC)  
February 1982 - January 1986

The purpose of this investigation is to develop and test (in a limited sense) models of electrodynamic processes involving liquids with poorly ionized solutes at high (applied) field strengths.

Extant theories which account for the details of the physicochemical processes associated with charged interfaces deal with mainly with low field strengths and fully ionized solutes. The model used to describe processes at high field strengths -- the leaky dielectric -- omits consideration of electric double layers, adsorption at interfaces, and chemical processes involved in the dissociation and recombination of solute species. Thus, even though the model depicts some features associated with bulk fluid motion faithfully, it fails to give a comprehensive picture.

The research involves several tasks: (1) construction of a mathematical model for low field strength electrokinetics for rigid particles with poorly ionized solutes, (2) construction of a similar model for fluid globules wherein the interface is permeable to ions, and (3) extension of the model described in (2) to high field strengths.

Design, Fabrication and Testing of an Experimental Apparatus  
for Determining Particle Behavior under very Low  
Intergranular Stresses

University of Colorado, Boulder  
Dr. Stein Sture  
Dr. Nicholas Costes, NASA/MSFC  
NAS8-35668 (NASA Contact: V. Yost, MSFC)

The objective of this research is to investigate the constitutive behavior of granular materials under very low intergranular stress conditions.

Research tasks include the design, fabrication and testing of an experimental apparatus for determining particle behavior under very low intergranular stresses. A dynamic response study of the Mechanics of Granular Materials (MGM) specimen and support assembly was conducted at a 1-g level in a geotechnical centrifuge in order to ascertain the integrity of equipment and specimen during liftoff and landing phases.

Presentations

Costes, N. C., "Microgravity Experiments on Granular Materials," to be presented at Materials Research Society Symposium on Materials Processing in the Reduced Gravity Environment of Space, Boston, December 1986.

Costes, N. C., "Mechanical Behavior of Cohesionless Granular Materials at Very Low Intergranular Stresses," to be presented at AIAA 25th Aerospace Sciences Meeting, Reno, January 1987.

## **D. FACILITIES**

## Microgravity Materials Science Laboratory

NASA Lewis Research Center  
Leslie A. Greenbauer-Seng  
In-House  
February 1984 - continuing task

The Microgravity Materials Science Laboratory (MMSL) was opened at the NASA Lewis Research Center in September 1985. The laboratory contains functional duplicates of space shuttle flight hardware, new experimental equipment concepts, and supporting equipment and instrumentation to study metal and alloy solidification and crystal growth. The MMSL is available for use by scientists and engineers from industry, university, and government agencies interested in studying materials processing in a low gravity environment. The laboratory is a 1-g research facility where investigators may take the first step toward defining key space flight experiments for the space shuttle and eventually the space station. The MMSL may also be used in preparation for experiments to be conducted in other ground-based facilities such as drop towers and research aircraft. Extensive materials characterization, computational, and machining/fabrication capabilities are readily accessible to the visiting investigators conducting experiments in the MMSL.

The experimental equipment which is now available for use in the MMSL include the General Purpose Furnace, Electromagnetic Levitation Furnace, Instrumented Drop Tube (1 sec), Undercooling Furnace and Dendrite Growth Apparatus. Functional duplicates of the Automated Directional Solidification Furnace and the Single Axis Acoustic Levitation Furnace will be available in 1986. In-house solidification and macrosegregation studies have led to the design of new experimental equipment to investigate novel processing concepts for space experimentation. Two NASA Lewis designed Directional Solidification Furnaces and a Bulk Undercooling Furnace are currently being developed to support this work. New MMSL capabilities will soon be added to support research of ceramic, glass, and polymer processing techniques in low gravity. Laboratory site preparation and procurements of instrumentation to support these materials areas have been initiated.

Numerous presentations, tours, and technical discussions with various industrial and academic representatives have been conducted throughout the year. An information booklet describing the experimental capabilities and the MMSL application process was printed and is continuing to be widely distributed to potential MMSL users.

Space Station Microgravity Science Accommodation  
Requirements Study

Wyle Laboratories

Mark L. Uhran

NAS3-24654 (NASA Contact: R. J. Parker, LeRC)

The objective of this task is to define, from the user/researchers' perspective, the accommodation requirements imposed on Space Station for performing microgravity science and materials processing experiments.

The study will search out and assemble input from users in microgravity science discipline areas including combustion science, electronic materials, metals and alloys, fluid physics and transport phenomena, glasses and ceramics, and polymer science. Basis for the requirements comes from an ongoing LeRC program in microgravity science and applications in this broad range of scientific disciplines and its concomitant participation and interaction of a large body of researchers. This close and continuing relationship with researcher's in both ground-based and flight experiments is used as a basis for achieving the needed emphasis on researcher's perspective for defining users' requirements for Space Station microgravity science and applications experiments.

Specific goals of the study are to (1) provide accommodation requirements input to the Microgravity and Materials Processing Facility (MMPF) study managed by MSFC and (2) provide an assessment of the needs for and plan for development of new experimental apparatus and supporting equipment for microgravity science experiments.

The first goal was achieved in December 1985 with the submittal of accommodation requirements input to the MMPF study for inclusion in the January 1986 data release to the Space Station Work Package #1 Phase B Contractors. User requirements, focused on the discipline areas of LeRC interest and expertise, have been identified in eleven research classes, each of which contain an envelope of functional requirements for related experiments having similar characteristics, objectives, and equipment needs. For each research class, utility, environmental, logistics, and safety requirements were identified.

Based on these functional requirements, seventeen items of experiment apparatus have been identified to accommodate these research classes. Likewise, twenty items of core equipment have been identified which represent supporting equipment required by nearly all of the research classes and user discipline groups.

An equipment development plan will be prepared following the assessment of the availability and technology readiness of the items of experimental apparatus and supporting equipment defined by the functional requirements.

**E. INDUSTRIAL AFFILIATIONS**

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## Physical Vapor Transport of Organic Solids

3M Company  
Dr. Mark K. Debe  
Dr. Earl. L. Cook  
Joint Endeavor Agreement  
(NASA Contact: James Fountain, MSFC)  
November 1984 - November 1986

The objectives of this research are: (1) to study the physical vapor transport process under microgravity conditions; (2) obtain thick, highly ordered crystalline films on selected substrates of sublimable organic materials; and (3) to proof test the experiment apparatus design for performance under microgravity conditions.

The 3M Physical Vapor Transport of Organic Solids (PVTOS) payload is an organic materials processing experiment designed for an Orbiter middeck installation. PVTOS is an experiment method for growing crystalline solids and films by gaseous diffusion. In this approach a source material is heated in one end of a reactor tube causing it to diffuse through another gas and then recondense on the cooler end of the tube.

There are two main components to the payload hardware, i.e., the experimental apparatus container or EAC, and the generic electronics module, or GEM. Inside the EAC are nine identical experimental cells and various electronic circuit boards specific to PVTOS. The individual cylindrical cells, each roughly 3 inches in diameter and 12 inches long, contain a vacuum insulated heater core surrounding a specialized reactor tube, a heat pipe cooled substrate within the reactor tube and thermocouples to monitor the substrate temperature and reactor tube hot end temperature. The cell design intrinsically provides double hermetic confinement of the source material utilizing all metal to metal seals.

The GEM provides all the control electronic functions for PVTOS, and as its name implies has been designed to control many other payloads as well. It contains a microprocessor with bubble memory and provides not only the interface between the Orbiter and the EAC, but also the crew and the EAC by featuring a hand-held keyboard and liquid crystal display terminal. Using this terminal the mission specialist can select and activate the cells, monitor the cell temperature and power levels, and perform diagnostic tests if necessary.

The PVTOS experiment is designed to yield results which have primarily scientific value and will most directly impact a 3M program on ordered organic thin films. Both



knowledge of thin film growth and the physical vapor transport processing a microgravity environment will result.

#### Publications

Debe, M. K., "Industrial Material Processing Experiments Onboard the Space Shuttle Orbiter," J. Vac. Sci. Techn., 1986 (in press).

## Diffusive Mixing of Organic Solutions: DMOS-1

3M Company  
Dr. William Egbert  
Diana J. Gerbi  
Dr. Earl L. Cook  
Dr. Christopher J. Podsiadly  
Joint Endeavor Agreement  
(NASA Contact: Dr. R.J. Naumann, MSFC)  
November 1984 - November 1986

The objective of this investigation is to evaluate the growth of various organic crystals by recrystallization from organic solvents, formation of insoluble organic salts, or formation of an insoluble product in a chemical reaction.

Six experiments were flown on STS-51A. Three of the cells contained proprietary materials. One cell contained urea dissolved in methanol, with toluene as the precipitating nonsolvent. Two of the cells contained a cationic cyanine dye and anionic oxonol dye, but a hardware malfunction prevented formation of the insoluble dye-dye salt. Initial characterization of the product materials showed no chemical or crystallographic structural differences between the microgravity-grown materials and one-gravity control samples.

A representative sample of the urea crystals (microgravity-grown and one gravity control) has been analyzed for size distribution. There is a tendency for the formation of long (10 mm long x 2 mm wide) single crystals in microgravity, while the one gravity control is characterized by polycrystalline clumps of more uniform aspect (3-4 mm on a side). Optical quality of a typical microgravity crystal is considerably better than that of a comparable one gravity control. Experiments are now planned to quantify the physical size and distribution of the light scattering defects in representative samples from each distribution. The size distribution will be further analyzed by NASA and compared to a model under development by Baird and Naumann.

Similar differences in crystal size and optical quality are observed in one of the proprietary materials. In microgravity the crystals form as free or attached clumps of single crystals ranging up to 10 mm long. In one gravity most of the material forms as a polycrystalline mat, with small (100 micron wide x 5 mm long) crystallites nucleating from points within the mat.

## Publications

Egbert, W. C., Gerbi, D. J., Ender, D. A., and Cook, E. L., "Nonlinear Optical Crystal Growth in Microgravity," in Proceedings of Symposium on Nonlinear Optical Materials, MRS, Boston, 1985 (Abstract).

Gerbi, D. J., Egbert, W. C., and Cook, E. L., "Growth of Organic Crystals in a Microgravity Environment," J. Cryst. Growth, 1985 (submitted).

Diffusive Mixing of Organic Solutions -2

3M Company  
Dr. Marc D. Radcliffe  
Dr. Earl L. Cook  
James Steffen  
Dr. Michael Runge  
Lowell Miller  
Joyce Cutting  
Dr. Christopher Chow  
Dr. Christopher Podsiadly  
Joint Endeavor Agreement  
(NASA Contact: James Fountain, MSFC)  
November 1984 - November 1986

The objective of this research was the investigation of the fluid mixing and crystal growth process of organic solutions in the microgravity environment.

Solutions of organic compounds contained within six hermetically sealed cells were allowed to mix during the orbit of STS 61-B. Four of these experiments produced crystals of 3M proprietary materials and a dye-dye salt of interest to 3M materials research. Two other experiments addressed the mixing behavior of the solutions as exposed to the accelerations present in orbit. The results obtained will be used to further 3M ground-based research, and to better understand the microgravity effect on materials processing.

**F. FOREIGN GOVERNMENT AFFILIATIONS**

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## Mercury Iodide Crystal Growth

Laboratoire de Cristallographie et de Physique  
Dr. Robert Cadoret  
Dr. Pierre Brisson

The objective of this research is to investigate the effects of weightlessness on vapor transport of single crystals of mercury iodide.

High quality mercury iodide crystals are very sensitive X-ray and gamma ray detectors that operate well at room temperature. Since comparable high-performance detector materials require extremely low operating temperatures, mercury iodide is an attractive material for a variety of uses in science and industry.

A standard technique for producing these crystals is by solidification from a vapor under carefully controlled growth conditions. In a closed container, vapor from a heat source material moves by slow convection and condenses upon a cooler seed crystal to produce a larger crystal. Crystal quality depends on constant temperature, growth rate, and vapor transport conditions. It is difficult to produce sufficiently high-quality crystals on Earth because small variations in pressure during the growth process create defects that impair the electronic performance of the crystals. These variations may be induced by gravity. Scientists are interested in understanding the effects of gravity on vapor transport so they can learn to control crystal growth conditions and thus control crystal quality. The practical result of this knowledge will be the production of improved materials for use as radiation detectors.

Evaporation and condensation will occur within ampoule cartridges in a two-zone furnace having different temperatures at each end. The experiment apparatus includes two heat pipe furnaces, each holding three cartridges, and six ampoule cartridges enclosing mercury iodide seed crystals and source material.

The experiment was performed on both Spacelab 1 and Spacelab 3. However, on Spacelab 3, the samples were subjected to different temperatures and growth conditions for comparison with the crystals previously grown on Spacelab 1. Reflight of the investigation demonstrates the capability of changing and repeating experiments on successive Spacelab missions. In this way, scientists can respond fairly rapidly to experimental results from research in space.

The three experiments performed in Spacelab 1 evidenced an unexpected effect of gravity on nucleation and growth of  $\text{HgI}_2$  crystals. This effect probably arises from concentration fluctuations due to local convections most especially during establishment of the stationary regime. Such local convections probably exist in the diffusion layer surrounding a growing crystal. The advantage of space is to reduce these concentration fluctuations and then to have an easier control of the quality of growing crystals. These experiments as well as the experiments prepared for Spacelab 3 were conceived in order to have a better understanding of the gravity effect on nucleation and growth.

#### Publications

Cadoret, R., Brisson, P., and Fries, E., "Effect of Gravity on Nucleation and Growth of  $\text{HgI}_2$  Crystals from Vapour," J. Cryst. Growth, 1986 (submitted).

Aggregation of Red Cells ("ARC"): A Study of Blood Flow and of Aggregation of Blood Cells Under Conditions of Zero Gravity - Its Relevance to Occlusive Diseases and Cancer

University of Sydney, Department of Medicine  
Dr. Leopold Dintenfass  
MPS77F113  
January 1985 -

The objectives of this project are: (1) to determine whether the size of red cell aggregates, and kinetics and morphology of these aggregates are influenced by near-zero gravity; (2) whether the actual shape of the red cell changes under near zero gravity; (3) whether viscosity, especially at low shear rates, is affected by near zero gravity; and (4) whether blood samples obtained from different donors (normals and patients suffering from different disorders) react in the same manner to near-zero gravity.

Actual findings during experiments on STS 51-C include: (1) the red cells do form aggregates under near-zero gravity, although these aggregates are smaller than corresponding ones obtained on the ground (in identical blood samples, under analogous functional conditions; (2) red cells did not change shape under near-zero gravity; (3) viscosity appears to be lower under low shear due to the findings described under (1), but a repeat of the experiment is necessary in order to define changes in viscosity exactly; and (4) blood samples obtained from donors showed different pattern for normal blood and for patients blood. Red cells in normal blood formed on the ground rouleaux (like pearls on a string) while a random pattern was observed under near-zero gravity. Red cells in blood of patients with ischaemic heart disease and cancer of the colon showed normal aggregation pattern under near zero gravity, while on the ground a sludge (compact) and heavy aggregation was noted. Red cells obtained from a patient on insulin (juvenile diabetes) showed lighter aggregation under near zero gravity, but these patterns should be reinvestigated. Platelets studied under near zero gravity appeared to form non interconnected swarms, retaining their original shape, and lack of pseudopods or tentacles characteristic of aggregation on the ground.

A preliminary conclusion, subject to confirmation on the subsequent shuttle flight is: (1) that near zero gravity affects cell-to-cell interaction; (2) that near zero gravity affects microstructure of the membrane of red cells and membrane of platelets; (3) that a question should be asked whether electrodynamics of cell membrane, and/or internal organization of cell interior are affected by near zero gravity; and (4) that a speculation could be explored



whether near zero gravity affects physicochemical or biochemical reactions.

#### Publications

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Dintenfass, L., "Red Cells under Zero Gravity," Lancet 1, 747-748 (1985).

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Dintenfass, L., "Influence of Plasma Proteins on the In Vivo and In Vitro Rheological Properties of Blood," Clinical Hemorheology 5, 181-206 (1985).

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Dintenfass, L., "Editorial: Red Cell Rigidity, "Tk", and Filtration," Clinical Hemorheology 5, 241-244 (1985).

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Dintenfass, L., Osman, P., and Jedrzejczyk, H., "Experiment on the Space Shuttle Flight 51-C: Aggregation of Red Blood Cells in Disease," Austr. Phys. Engr. Sci. Med. 8, 110-115 (1985).

Dintenfass, L., Osman, P., Maguire, B., Jedrzejczyk, H.,  
Jedrzejczyk, B., and Edwards, D.K.G., "Results of  
'Aggregation of Red Cells' (ARC) Experiment on Space Shuttle  
'Discovery' STS-51C on January 24/25 1985," in Proceedings  
of Second National Space Engineering Symposium, Sydney,  
March 1986.

MEPHISTO - Interfacial Destabilization and Segregation in Metal Alloys and Semiconductors

Center for Nuclear Studies of Grenoble  
Dr. J. J. Favier  
Dr. D. Camel  
Dr. A. Rouzaud

The objective of this investigation is to study the destabilizing mechanisms at a solidification interface as well as segregation behaviors in order to obtain information on the kinetics and morphologies in the transient and steady-state, and to separate the influences of liquid phase instabilities from interfacial instabilities.

Conceptual design of the experiment is complete. Sn-0.1A%Bi to Sn10%Bi and Bi-0.01A%Sn to Bi-1%Sn will be directionally solidified at rates varying from 0.0001 to 0.1 cm/sec with thermal gradients varying from 10 to 400 K/cm. A differential Seebeck voltage measurement technique has been developed to provide a continuous record of the solid-liquid interface temperature as the solidification rate is varied in order to determine the kinetic coefficients. Signal processing and noise suppression techniques have been demonstrated to allow nanovolt precision which corresponds to mK accuracy for the interfacial temperature.

The investigation currently underway include work on macrosegregation and directional solidification of doped and concentrated alloys and semiconductors with special care to the coupling between the hydrodynamics of the melt and the growth phenomena at the interface. An order of magnitude analysis is developed to define the boundary layers and scales laws as a function of the boundary conditions. Morphological stability theory is also reconsidered in the frame of deformable boundary layer assumptions. These concepts are checked on the ground by using appropriate furnaces with compensated radial thermal gradients. Seebeck measurements of the solid-liquid interface temperature permit to check the scaling laws for mass and heat transfers as well as to quantify the bifurcation of the various oscillatory models into turbulence (Fourier analysis). This method will be useful in space to check the influence of g-jitters on the solidification process.

At last dendritic growth is also studied and the influence of the fluid flows of different origins (bulk and interdendritic pools, thermal or solutal) is predicted. A first set of experiments on dendritic solidification in the Al-Cu system and cellular growth in Pb-Tl has recently been flown on the D-1 mission.

## Publications

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## Directional Solidification of Monotectic and Hypermonotectic Aluminum-Indium Alloys under Microgravity

Center for Nuclear Studies of Grenoble  
Dr. Claude Potard  
Dr. Paul Morgand

The objective of this program is to analyze the mechanisms involved in the composite solid structure formation obtained from a miscibility gap alloy under microgravity. The metallic system Aluminum-Indium has been chosen for its low critical temperature, broad miscibility gap, and rather well-known thermodynamic properties.

Two previous experiments were performed on this subject.

The first was the sounding rocket flight SPAR IX: A CEA-NASA joint action which took place on January 21, 1981.

The second one, a collaboration CEA-SOVIET UNION, was performed aboard the Salyut-7 spacecraft (June-July 1982).

The MEA-A3 experiment results from a collaboration CEA-NASA. It is scheduled to fly in July 1986. Its objective is to elaborate a hypermonotectic, immiscible Al-In alloy in microgravity from a melt heated above the miscibility gap. This run was designed so as:

- To keep a very low level of thermal gradient in the liquid metal in order to minimize the capillary effects (migration of In droplets and convection).
- To provide a planar, directional solidification front.
- To minimize the free volume effects.
- To force the In droplets into incorporation with the S/L front.

Those last two points make the difference between the MEA-A3 experiment and the previous ones.

The theories of capture of particles by an advancing front were not able to explain the observations made on SPAR and Salyut-7 experiments. A new model was developed based on the filtration theory considering the globules population: the solid/liquid interface is like a filter of negligible thickness for the globules and its efficiency is limited by a pressure barrier measured for Al-In alloys by ground based experiments. The MEA-A3 experiment was consequently adjusted to take into account the new model.

The behavior of the globules in an interacting population on the solid front is, of course, an essential factor in the theory. The theory is at present being completed by results obtained by the Laboratoire d'Aerothermique of Meudon. The hydrodynamic and thermal interactions between pairs of globules placed in a temperature gradient have been calculated showing a large reduction of the Marangoni effect compared to the isolated globule. Physico-chemical properties of metallic liquid interface are currently being investigated within a cooperative program between CNES and the University of Grenoble.

#### Publications

Morgand, P. and Potard, C., "Immiscible Alloys, Experiment Aboard Salyut-7: Results and Interpretation of Aluminum-Indium Emulsions Solidified in Microgravity," Adv. Space Res. 4, 54 (1984).

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16. Abstract <p>This report is a compilation of the active research tasks as of the end of the fiscal year 1985 of the Microgravity Science and Applications Program, NASA-Office of Space Science and Applications, involving several NASA centers and other organizations. The purpose of the document is to provide an overview of the program scope for managers and scientists in industry, university, and government communities. The report includes an introductory description of the program, the strategy and overall goal, identification of the organizational structures and people involved, and a description of each task. The report also provides a list of recent publications.</p> <p>The tasks are grouped into six major categories: Electronic Materials; Solidification of Metals, Alloys, and Composites; Fluid Dynamics and Transport Phenomena; Biotechnology; Glasses and Ceramics; and Combustion. Other categories include Experimental Technology, General Studies and Surveys; Foreign Government Affiliations; Industrial Affiliations; and Physics and Chemistry Experiments (PACE). The tasks are divided into ground-based and flight experiments.</p>			
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